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TEXTILE RESEARCH JOURNAL

The Surface of Cotton Fibers

Part IV: Distribution of Dry Soil

Verne W. Tripp, Anna T. Moore, Blanche R. Porter, and Mary L. Rollins

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Abstract

The location of soiling agents such as colloidal and activated carbon, floor soil, a synthetic soil, and a clay on gray and modified plain weave cotton fabrics soiled under controlled conditions was determined by light and electron microscopical examination. Gross and fine geometry of fabric and fiber play a major role in determining the distribution of soil in most cases. The mechanical entrapment of soil particles and agglomerates between fibers near the surface of the fabric is repeated in the accumulation of soil in the natural or induced irregularities of the native and modified fiber surfaces. The presence of soft films on the fiber surface extends the quantity of soil and its distribution. Sorption of the soil particles on apparently smooth areas of the fiber also occurs to a significant extent.

METHODS for the evaluation of soiling of textiles and the development of means for improving the soil resistance of such materials have received considerable attention in the past few years. In such studies, estimates of the soil uptake of the textile have been carried out by reflectance, turbidimetry, chemical analysis, radioactive tracer assay, and other means [2, 6, 7, 9, 10]. Microscopy also has been used in several investigations [3, 5, 8, 9] primarily as a tool for locating and examining the soiled areas of the textile material. In conjunction with a survey of the topography of native and modified cotton at this laboratory, a microscopical study of the distribution of soil on fiber surfaces was carried out. Since many types of soiling are limited to the surface regions of textile structures, soiling may be considered to be a special case of the additive finishing of fibers. Many soiling agents contain solubilized components which can be carried to the interior regions of textile fibers by liquid media, but the attachment of solid soil particles from nominally dry sources is also an important part of the overall soiling phenomenon in textiles. The present study was limited to the examination of such "dry soil" types on the surface of cotton textiles and relates to the initial soiling of a clean fabric surface. It does not include observations on soil redeposition, as in washing, which is discussed elsewhere in the literature [1].

[1] One of the laboratories of the Southern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture.

Experimental

Soiling Methods

The soiled cotton fabrics examined were prepared by methods described in detail in the previous paper in this series [6]. The soiling methods included: (a) foot traffic on the fabrics in the corridors and storerooms of this laboratory; (b) tumbling of the fabrics in a rotating cylinder with a mixture of glass beads and a known amount of such soils as colloidal carbons of varying primary particle sizes, finely ground activated carbon, a ceramic clay, and a synthetic soil made up according to the formula of Salsbury et al. [8]; and (c) on a limited number of fabrics, electrostatic attraction of sprayed carbon particles by giving vertically hung fabric samples a positive charge of approximately 1000 volts. Prior to examination, after soiling, all specimens were thoroughly vacuum cleaned, using the upholstery attachment of a commercial tank type cleaner.

Samples

The soiling response of the plain weave fabrics studied microscopically was noted in the previous paper of this series [6]. Gray, bleached, and mercerized fabrics were examined, as well as representative chemically modified cottons. The chemical modifications included partial acetylation, partial cyanoethylation, partial carboxymethylation, and impregnation with melamine-formaldehyde resin. The observations also covered fabrics with additive finishes, such as starch, carboxymethyl cellulose, colloidal silica (Ludox),[2] Juvenon R,[2] acrylic polymer latices, a silicone resin, a dialkyl ketene, and perfluorocarboxylic acids and their derivatives. In most cases the fabrics were of 80 × 80 print cloth construction.

Inspection Methods

The fabrics were examined at low magnification by oblique illumination in order to determine the overall distribution of soil with respect to gross fabric structure. Yarns removed from a number of the fabrics were observed in cross section. Single fibers removed from the fabric yarns also were examined at high magnifications with the light microscope. However, most attention was given to the appearance by electron microscopy of surface replicas of soiled fibers prepared directly from the fabric specimens. The techniques involved are described in the first paper of this series [11].

[2] Mention of trade names does not imply endorsement of the product over similar products of other manufacturers.

Results and Interpretation

Fabrics

Inspection of fabrics soiled by the three methods mentioned above showed that differences in the gross distribution of soil were present. The surface of fabrics treated by the tumbler method were soiled most evenly, while those subjected to floor traffic

Fig. 1. Cross section of yarn removed from 80 × 80 fabric soiled by tumbler method with synthetic soil. 250 ×.

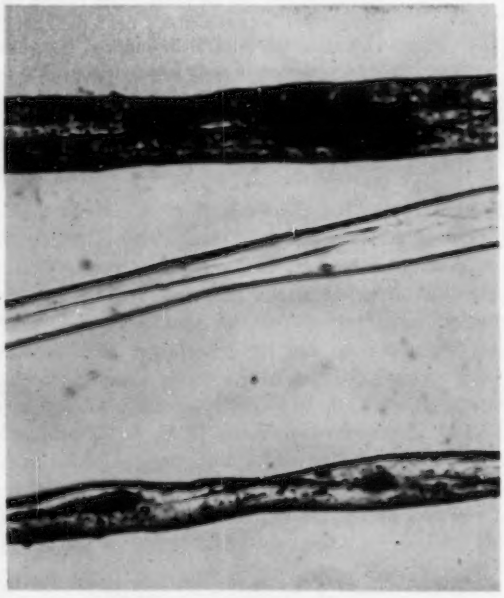

Fig. 2. Longitudinal view of an unsoiled fiber and two fibers soiled with synthetic soil. 500 ×.

exhibited large variations in soil pickup from one area to another. When electrostatic attraction was employed, an even deposition of soil occurred, but the carbon particles were limited almost exclusively to the fibers projecting up from the plane of the fabric surface. Soil deposited by this method was almost completely removed by vacuum cleaning. As would be expected, heavy soil deposits occurred on the high points of the fabric yarns in both tumbler soiled and floor soiled specimens. Much of this soil was held between fibers of the yarns, being mechanically trapped by the opening and closing of spaces between fibers when pressure was applied to the fabric during the soiling process.

Yarns

In none of the fabrics was there evidence of deep penetration of the soil into the yarn structure. The surface regions in areas of contact between yarns were virtually unsoiled. The typical appearance of a soiled yarn cross section is shown in the photomicrograph in Figure 1. The fabric from which this yarn was taken had been heavily soiled with synthetic soil by the tumbler method. The dark areas indicating the location of soil are limited largely to one side of the yarn. The soil does not extend beyond the first few fibers of the yarn periphery. This picture is also representative of yarns removed from fabrics soiled by foot traffic.

Fibers

Examination of individual soiled fibers taken from yarns showed considerable variation in the amount of soil on each fiber and from point to point on a single fiber. Figure 2 shows short lengths of three fibers, two of which had been soiled with synthetic soil by the tumbler method in a fabric; an unsoiled fiber is included for comparison. The upper fiber is heavily soiled in most areas, while the lower shows some regions which are relatively clean. The aggregation of soil in the relatively large concavities present in every fiber is apparent, and has been commented on by Hall [4] and Compton and Hart [1]. At many points on the fibers, however, soil is distributed on apparently level regions.

In the preparation of surface replicas of soiled fibers by the two-stage method [11], the final "positive" replica which is examined in the electron microscope may be termed a "pseudo-replica." In such replicas, a portion or all of the original surface is removed from the specimen and carried through to the final replica. In this case, the soil particles and aggregates present on the fiber were transferred and observed in the microscope. Virtually complete transfer of the soil from fiber to replica was evident when the fiber or fabric specimen was stripped from the polystyrene film used for obtaining the initial impression. After stripping, the soiled fibers were almost completely clean. From this observation as well as a comparison of the appearance of soiled fibers and the replicas obtained, it is concluded that the replicas give a faithful representation of the location of the soil on the fiber surface.

Characteristic appearances of the surface of unscoured fibers in soiled fabrics are shown in Figure 3. Three different soil types are represented. In the micrograph at the left, colloidal carbon whose primary particle size was 450 Å was the soiling agent. It was applied by the tumbler method. The center micrograph shows the distribution of particles of an activated carbon (average particle size 1000 Å), also applied to the fabric by the tumbler method. At

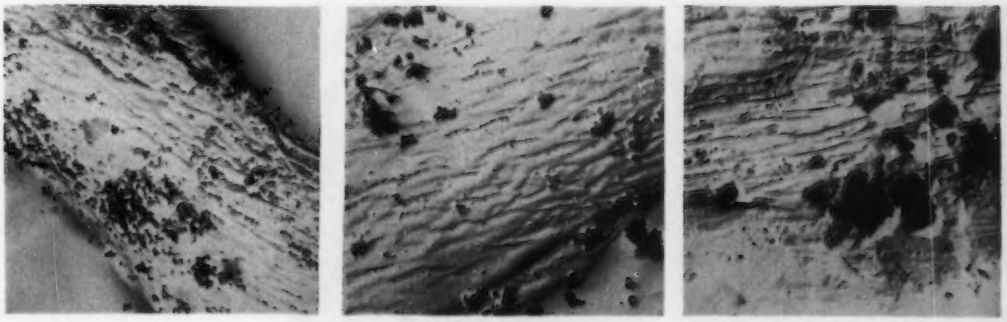

Fig. 3. Electron micrographs of surface replicas of fibers in fabrics soiled with colloidal carbon (left), activated carbon (center), and floor traffic soil (right). 4000 ×.

right, the surface of a fiber in a fabric soiled by floor traffic is shown. In each micrograph, the characteristic pattern of ridges and grooves generally observed on the surface of most cotton fibers may be seen.

In the case of fibers soiled with colloidal carbon, there is strong evidence for preferential aggregation of the soil in the grooves of the fiber surface, although considerable soil appears to be held on relatively smooth areas also. Larger aggregates of soil may be built up by the soil initially held mechanically in the grooves serving as a nucleus for attachment of additional soil.

The picture of fiber surfaces soiled with the activated carbon is somewhat different. There is definitely less tendency for aggregation of the carbon particles, and the indentations on the fiber surface are seen to be filled with smaller particles only. Larger particles are seen clinging to other regions of the fiber surface. The attachment of the larger particles seems to be not so much a mechanical entrapment in the surface roughnesses, as is the case with the smaller sized carbons, but due to the physical shape of the particles themselves. The sharp points on the activated carbon particles may play an important role here. Fibers soiled with clay were similar in appearance to those soiled with activated carbon.

Soil from floor traffic is more heterogeneous in composition than the other soiling agents used, and a number of components present may be visualized. It is apparent that the component of small particle size has taken up a position in the grooves of the fiber surface. Other particles or agglomerates of soil, which are themselves too large to fit into the surface grooves, may be attached to the fiber through the material which fills the grooves. The synthetic soil applied to the fabrics by the tumbler method presented an appearance similar to that observed for the floor traffic soil.

The importance of surface roughness in promoting the buildup and concentration of soil on the fiber is illustrated in the two electron micrographs in Figure 4. The micrograph at the left shows a point on the surface of the fiber at which the direction of the ridges reverses itself. Such areas are themselves replicas of the reversals in the direction of spiral in the cellulose fibrils of the secondary wall of the fiber [11]. The pattern of soil accumulation on the fiber surface at this point follows the direction of the grooves to a noticeable degree. In the micrograph at the right, a similar phenomenon is noted. Here a number of transverse protuberances on the fiber surface have caused the buildup of relatively large aggregates of soil. Protuberances such as these are seldom seen in most cottons but are frequent in fibers which have been subjected to compression [11].

The previous report in this series [6] showed that there was a wide range in the extent to which soil was picked up by cottons treated with a number of typical finishing agents. In most cases, the appearance of the surface of the fibers soiled after such treatments did not differ markedly from that of untreated cotton which had been soiled in the same manner. Some surface finishes, however, gave strikingly different results. An example of such behavior is given in the micrographs of Figure 5. At left is the surface of a fiber from a fabric treated with a soft acrylic polymer latex emulsion. The add-on of polymer was approximately 30%. After soiling with colloidal carbon by the tumbler method, the typical surface appearance was that in the micrograph at the

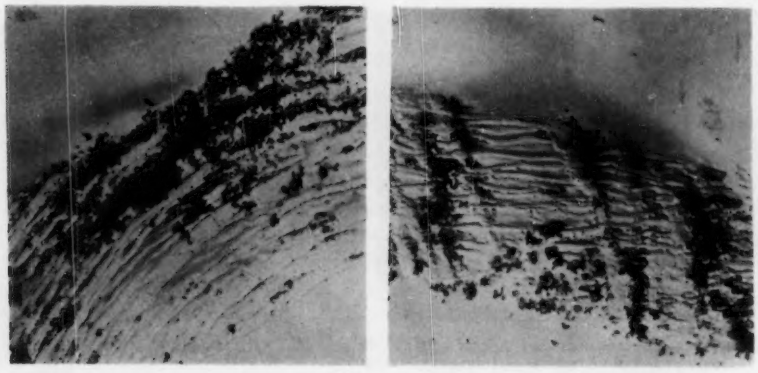

Fig. 4. Electron micrographs of surface replicas of fibers soiled with colloidal carbon. Left: accumulation of soil in patterns following ridges at reversal point in fiber. Right: accumulation of soil at transverse protuberances on fiber surface. 3000 ×.

right. Although the surface roughness has been reduced to a noticeable extent by the coating of coalesced polymer, the affinity of the new surface for soil is enormously increased. In such fabrics, the application of the vacuum cleaner had little or no effect on the appearance of the soiled areas. The mechanism of soil attachment in such cases is undoubtedly related to the relatively soft surface of the polymer coating [6, 12].

Among the finishes which improved the soil resistance of the cotton, the particulate types were found to be markedly effective [6]. In examining the surface of fibers which were soiled after these treatments, considerable difficulty was experienced in clearly differentiating the soil particles from the finish itself. Perhaps the clearest distinction that could be made was in the case of the specimens soiled with activated carbon, where the typical shape of the soil particles permitted identification. Figure 6 shows the surfaces of fibers from a fabric treated with Juvenon R [2] to give a 7% add-on before and after soiling with activated carbon. The small particles of the soil resistant finish cover the fiber surface rather completely. The larger, irregularly shaped carbon particles are distributed over the surface to a noticeably smaller extent than was typical for the untreated fiber (Figure 3, center). Other finishes, such as perfluorodecanoic acid [13] which gave significant resistance to soiling with little or no change in the surface geography of the fibers, showed a distribution of soil particles similar to, but of lesser extent than, untreated fibers.

Discussion

For a given soil–fiber system, the extent of soiling will depend on the combined results of the two successive phenomena of impingement and retention. In comparison to wet soiling, most dry soils are inherently more heterogeneous in their total composition and manner of application. Considered in this aspect, the distribution of dry soil on textiles will be uneven. Both impingement and retention will also be influenced markedly by the gross and fine geometry of the substrate. The impingement of

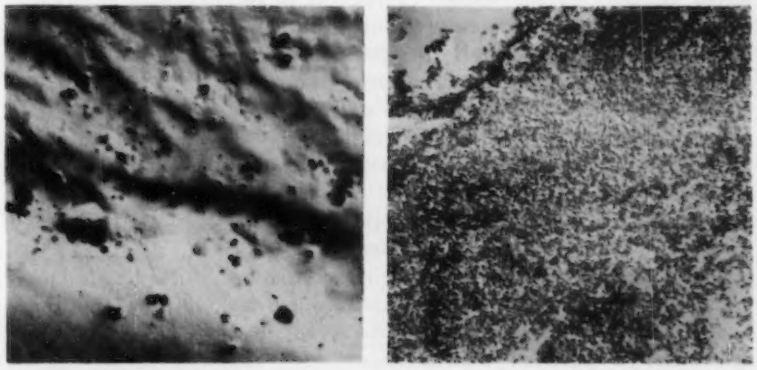

Fig. 5. Electron micrographs of surface replicas of fibers from fabrics treated with soft acrylic polymer latex emulsion. Left: before soiling. Right: after soiling with colloidal carbon. 8000 ×.

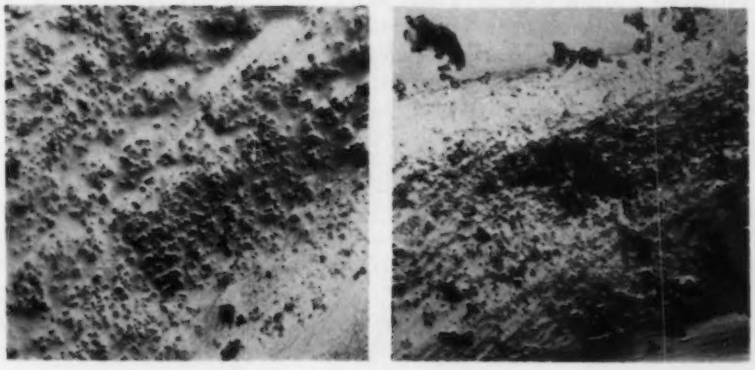

Fig. 6. Electron micrographs of surface replicas of fibers in a Juvenon R-treated fabric. Left: before soiling. Right: after soiling with activated carbon. 4000 ×.

soil particles attracted by electrostatic charge on the fabric soiled only those fibers which were immediately accessible; the relatively small force with which the soil was brought into contact with the fiber under these conditions was insufficient to cause penetration of the particles into the fabric structure or the irregularities of the fiber surface. In tumbler or floor traffic soiling, however, the forces of impingement are sufficient to cause intimate contact with and even deformation of the textile. Consequently, mechanical entrapment of the soil between fibers occurs and is repeated on the fiber surface, where roughnesses, concavities, grooves, and ridges serve to intercept and imprison the impinging particles. The particle size of the soil will also determine the probability of entrapment by various irregularities.

The observed location of dry soils on cotton makes it clear that fabric and fiber geometry governs the overall distribution of soil to a considerable degree. The natural irregularities of the fiber surface, which persist through modifications such as mercerization, partial acetylation and cyanoethylation, resin impregnation, and some types of additive finishing, are preferred locations for the buildup of soil. It is also evident that cohesion of more soil to that previously attached to the fiber occurs to a significant extent. However, considerable amounts of soil appear to be attached to the fiber surface in areas which are free from irregularities. Sorption of soil on the fiber surface independently of the more obvious mechanical entrapment thus is indicated also.

Insofar as microscopical observations could show, modifications of the fiber surface brought about by scouring, which eliminates the noncellulosic coating on the surface of cotton [11], or by carboxymethylation, which tends to roughen the surface [12], had little effect on the distribution of soils in these experiments. Application of a soft resin, which in effect permits deformation of the fiber surface and embedding of the soil particles, however, increases the soil retention and widens its distribution. The desirable effects of the particulate additives [6] on soil retention might be achieved not only through their physical occupation of potential sites of soil attachment, but also by their rendering the fiber surface less susceptible to deformation by the impinging soil under contact pressure incidental to soiling in many practical situations.

The sorption of soil may be decreased by lowering the surface energy of the fiber [13]. Mechanical entrapment is still operative under these conditions, but it receives little reinforcement from the van der Waals forces ordinarily present. Microscopy is of little avail in studying this aspect of soiling, but serves to indicate that the solution of the vexing problem of soiling probably lies in the field of physical chemistry as much as in fiber technology.

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The Variability of Set in Keratin Fibers

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Abstract

The amount of "permanent" set in a number of keratin fibers was measured, the fibers being strained 40% for 1 hr. in distilled water at 100° C. and then released slack for 1 hr. in the water. Variation in the amount of set retained by various keratin fibers was noted. The amount of set appears to correlate strongly with the ability of the particular fiber to supercontract and tends to be dependent on the fiber diameter.

Introduction

Speakman [4, 6, 7] and others have measured set in both wool fibers and hair under varying physical and chemical conditions. However, no attempt has been made to measure the change in the amount of set obtained under similar conditions for various fibers. Using fibers available at this laboratory, a limited survey has been made of the so-called one hour index of set in water as well as in 0.05 *M* borax solution. In the former case a great variability was found among both wool fibers from different sheep and other animal fibers.

We have limited our survey to the "permanent" set of fibers as described by Speakman [6] and have made no attempt to find out the variability of cohesive set among the different fibers.

Experimental Method

The fibers used in these experiments were all solvent degreased with cold petroleum ether. The ether was then washed away using cold ethanol and the fibers rinsed with cold tap water. Finally the fibers were washed in distilled water and allowed to dry.

To set the fibers, a stretching frame (Figure 1) capable of setting six fibers at a time was used. The fibers were formed into a loop with both the free ends held by one plug, and the loop over a hook. Each fiber was carefully adjusted so that the natural lengths of all six fibers in the frame prior to stretching were as close to equal as possible. The six fibers were extended simultaneously in water at room temperature (20° ± 1° C.) to the required setting strain. They were then boiled at this strain in distilled water for varying periods. At the end of the requisite period all strain was removed and the fiber loops were removed from the hooks, but the ends were still held by the plugs. The fibers were then boiled in a slack state in distilled water for 1 hr. The length of each looped fiber at the end of the hour was then measured; the strain remaining in the fiber was referred to as "permanent" set. When fibers were held strained at 40% extension for 1 hr. in boiling water and released slack in the boiling water for 1 hr., the measurement of "permanent" set was referred to as the "one hour index" of set for the fiber in water. This follows the terminology of Speakman.

The diameters of the fibers were measured at room humidity and temperature (55% ± 5% relative humidity and 20° ± 1° C. temperature), uniform fibers being chosen for test.

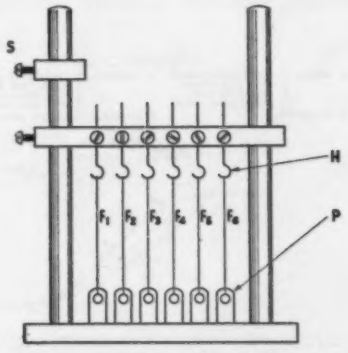

Fig. 1. Diagram of the draw frame for setting six fibers simultaneously. The fibers (F) are formed into loops over the hooks (H), and both ends of each fiber are held by a tapered plug at P. The stop (S) is adjusted to enable the correct setting strain to be applied to the fibers.

TABLE 1. Percent Set

	Corriedale SW 176, pengrown	Corriedale SW 1, fieldgrown
	21.8	11.8
	21.1	12.0
	20.6	13.2
	17.7	12.5
	20.3	10.7
	18.0	13.1
	19.3	12.5
	20.5	12.2
	20.9	
Average	20.0	12.3
Average diameter, μ	29.6	38.2

Results and Discussion

The Effect of Diameter on Set

Measurements of the one hour index of set in water were carried out on wool from ten different sheep. Also, tests were carried out on three lots of human hair and one lot of mohair. The readings were carried out on 6–10 single fibers in any one lot. Two typical sets of results for two different wool lots are shown in Table I. In Figure 2 the values of the one hour index for the different fibers are summarized with the corresponding average diameter (measured at room humidity and temperature) for these fibers. In general the one hour index fell into two distinct values and a transitionary value: (a) fibers with diameters below 30μ; (b) fibers with diameters about 50μ; and (c) fibers with diameters between 30 and 50μ having one hour indices varying roughly linearly with their diameter (see Figure 2).

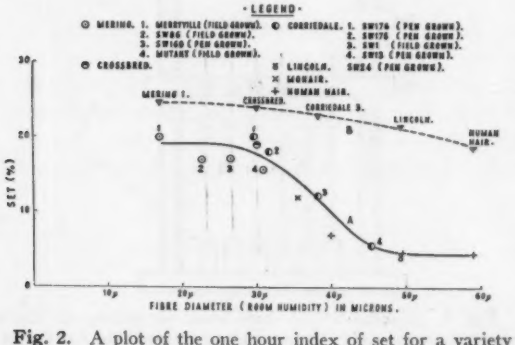

Fig. 2. A plot of the one hour index of set for a variety of fibers against the corresponding diameter of those fibers measured at room humidity. Curve A corresponds to fibers set in water and Curve B to fibers set in borax solution.

To obtain a clearer picture of the setting process, fibers were tested from each of the three following diameter ranges: (a) about 30μ; (b) about 40μ; and (c) about 50μ. These fibers were held strained in boiling water at 40% extension for varying periods of time, and released for 1 hr. in boiling water. The amount of set against time held extended is shown in Figure 3. When the amount of set is plotted against the logarithm of time (Figure 4), the three curves (A, B, and C) in Figure 3 reduce to three curves which are linear for up to a time of about 100 min., with the linear portions approximately parallel.

Consider an exponential relationship of the form

$$S = S_0 - S_1 e^{-t/\tau} \qquad (1)$$

where S_0, S_1, and τ are constants. When S is plotted against $\log_e t$, there results an approximately linear part of the curve for values of t in the vicinity of $t = \tau$ (see Figure 5). The slope of this linear portion is given by

$$\frac{dS}{d(\log_e t)} = \frac{S_1}{e} \qquad (2)$$

Changes in the value of τ in Equation 1 do not change the value of this slope S_1/e but merely move the curve of S against $\log_e t$ along the $\log_e t$ scale by an amount equal to the logarithm of the ratio of new to the original value of τ.

It follows from the above brief analysis that if Curves A, B, and C in Figure 3 are assumed to a first order approximation to be exponential, and with the same theoretical origin (i.e., at $t = 0$, the same value of set), the three parallel curves in Figure 4 have the same asymptote and differ only in time constant "τ." The time constant of C is about three times, and B about twice the time constant of Curve A.

Other one hour index tests, using Speakman's technique of setting in a 0.05 M borax solution and releasing in water, resulted in a percentage set (for five representative fibers chosen; Figure 1), which varied with diameter of the fiber, but less markedly than previously. In the 0.05 M borax the rate of setting is speeded up considerably (Figure 3) by about a decade compared with setting in water. Hence finer fibers reach maximum set in far less than 1 hr., and coarser fibers approach maximum set in this time. This results in a reduced difference in set among fibers of different diameters.

It is worth noting that the diameter range for wool fibers from 30 to 50μ corresponds to the transi-

tion range from bilateral distribution to radial distribution of ortho- and paracortices. A direct confirmation of any relationship between corticular structure and the amount of set has not been obtained.

Supercontraction and Set

It has been pointed out [5] that the mechanisms of supercontraction and set of keratin fibers are related. To check the relationship between set and supercontraction as a possible guide to the variability of set, the length to which a fiber fully supercontracted in concentrated lithium bromide solution (about 8 *M*) was compared with the one hour index of set for the same wool or hair. The comparison was done for wools and hairs for which figures were readily available in this laboratory. A more thorough comparison between these two mechanisms has been left to a later paper.

A direct correlation appears to exist between the amount a fiber supercontracts in a concentrated lithium bromide solution and the amount of set as measured by the one hour index. As a general rule, the coarser keratin fibers, such as human hair [1, 3], supercontract less than finer wool fibers under the same conditions. Iodination of the tyrosine group in wool, which inhibits the supercontraction of wool [2], also radically reduces the rate of setting of wool (see Figure 3). However, exposure to ultraviolet radiation under conditions which speed up the supercontraction process also increases the rate of setting (see Figure 3). To test further this idea that ability to supercontract in keratin fibers is related to the setting of these fibers, two special wools were chosen because of their low supercontraction in lithium bromide solutions:

1. SW10, a merino wool which supercontracted to 25%. Fine wools usually supercontract about 40% in 8 *M* lithium bromide solution. The diameter of this wool was about 22μ.

2. A high crimp merino wool which supercontracted 35%. The diameter of this wool was about 24μ.

In the first case, where the supercontraction was extremely low, the one hour index of set was about 3% instead of about 19%, as expected by the diameter of the fiber from Figure 1. In the second, the one hour index of set was 13–14%.

Clearly the relationship between the one hour index of set and diameter as expressed in Figure 2

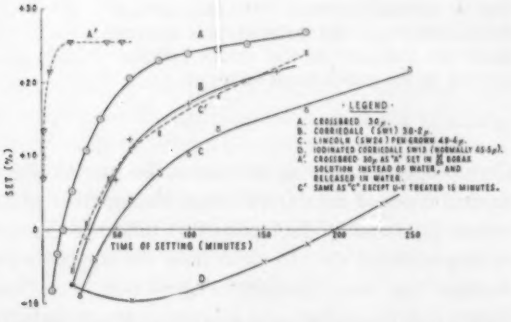

Fig. 3. Time of setting of various fibers plotted against percentage permanent set, showing the effect of a variety of treatments and diameters.

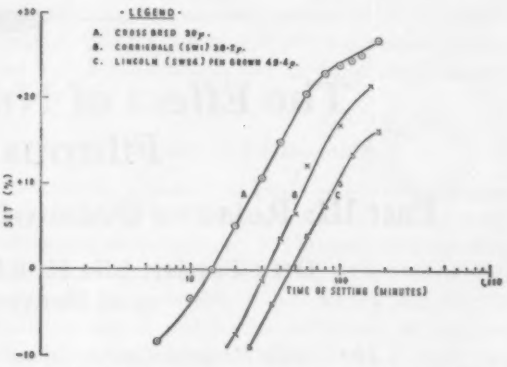

Fig. 4. The plot of percentage set against the logarithm of time of setting for the curves A, B, and C of Figure 3.

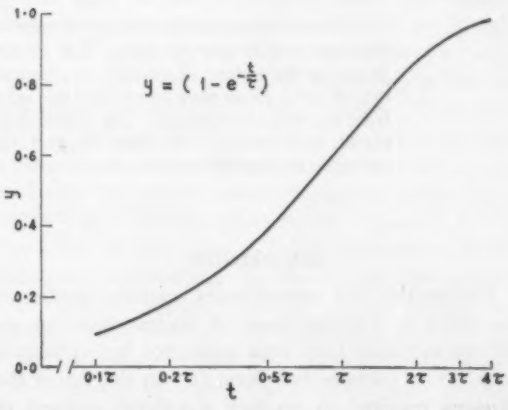

Fig. 5. The plot of y versus log t with the relaxation time τ as the unit of time "t" in the equation $y=(1-e^{-t/\tau})$ demonstrating the linearity of the relationship for t about τ.

is not perfectly general but applies to the normal run of wool and animal fibers. Special fibers can be chosen which fall well outside this relationship. The correlation between the ability of a fiber to set and to supercontract appears to be quite strong.

Conclusion

The ability of a keratin fiber to set is related to its ability to supercontract in a strong lithium bromide solution. Further, a variability from fiber to fiber in the time constant of the setting process (as indicated in Figure 3) exists which can be as great as 3:1. The coarser fibers have a strong tendency toward the longer time constant and the finer fibers toward the shorter time constant.

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The Effect of Nuclear Radiation on Fibrous Materials[1]

Part III: Relative Order of Stability of Cellulosic Fibers

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Abstract

Cotton, rayon, and acetate yarns were exposed for different periods of time to thermal neutrons and to gamma rays. The results have shown that total dose is the deciding factor in the extent of change in physical properties of the materials and that gamma rays (Co^{60}) alone have essentially the same effect as thermal neutrons when the energies from the two are equated. The decreasing order of stability of the materials was acetate, rayon, and cotton. The data suggest that there are linkages in the native cellulose molecule susceptible to radiation that are not present in the regenerated materials.

Introduction

During the past several years intensive studies of the effect of different kinds of nuclear radiation on fibrous materials have been conducted in this laboratory. The purpose has been (a) to determine the dosages required to produce significant changes in fiber properties, (b) to determine the rate at which several types of nuclear radiation change fiber properties, (c) to study the factors that influence the rate of degradation, (d) to determine the approximate dosages that cause complete degradation of various fibers, (e) to estimate the relative order of stability of fibrous materials, and (f) to determine insofar as possible the mechanism of degradation of fibers of

[1] Presented at the spring meeting of The Fiber Society, Clemson, S. C., April 30, 1958.

various chemical types. Consideration is given in the present paper principally to the relative order of stability of cellulosic materials,[2] these being the chemical types that exhibit rather rapid changes under the influence of nuclear radiation. As a minor but important consideration, the effects of two dose rates and of different types of radiation were investigated.

Materials and Methods

The materials used in this work were Deltapine cotton of known history spun into 30's yarn, 1100/480 high tenacity rayon yarn, and 100/25 cellulose acetate yarn. All were wound into small skeins, folded several times, and wrapped in thin, pure aluminum foil for exposure.

All samples were exposed to four sources of radiation at the Brookhaven National Laboratory. For neutron bombardment a water-cooled exposure port (Facility W-52) and the instrument tunnel in the reactor were used. The water-cooled exposure port had an estimated thermal flux of 1.5×10^{12} neutrons/cm.^{2}/sec. Samples were exposed in this facility for 13 different periods of time from 1 to 1000 min., thus receiving a total flux ranging from 9×10^{13} to 9×10^{16} neutrons/cm.^{2} The instrument tunnel had an estimated flux of 2×10^9 thermal neutrons/cm.^{2}/sec. Samples were exposed from 1 to 70 hr.; the total flux thus received ranged from 7.2×10^{12} to 5.0×10^{14} neutrons/cm.^{2}. The gamma irradiations were carried out using two Co^{60} sources, Facility 34 and the underwater 2-in. tube, having dose rates of 5.0×10^5 and 4.1×10^4 roentgens/hr., respectively. The samples in Facility 34 were exposed for 0.22, 0.70, 1.02, 10.0, 21.0, and 60.0 hr. to achieve total doses of 1.08×10^5 to 3.0×10^7 roentgens. Samples in the underwater tube were irradiated for 2.6, 5.8, 25.5, 122.0, and 265 hr. Total doses here ranged from 1.05×10^5 to 1.05×10^7 roentgens.

It is noted in the case of neutron irradiation that the total doses the samples received at the two flux rates overlapped only in the lower levels of exposure. It was not practical, even though desirable, to carry out exposures for longer than 70 hr. at the low flux rate. In the instance of gamma irradiation, however, there was a wide range over which the total dosages at the two rates corresponded.

[2] In the discussion in this paper, for the sake of convenience in writing, cellulose acetate yarn is referred to as a "cellulosic" material rather than a cellulose derivative or a cellulose ester.

The Instron Tester was used for the strength-elongation determinations. Tenacity, elongation, and average stiffness were evaluated from these data at break, while the modulus of elasticity was calculated at the yield point. Viscosity measurements were made in Ostwald-Fenske viscometers at 25° C. ± 0.025° C. Cupriethylene diamine (0.5 *M*) was used as the solvent for cotton and rayon, 0.50% and 1.0% solutions being used, respectively. The cellulose acetate samples were first extracted in benzene to remove processing oils and, after drying, the viscosity of a 0.50% solution of the acetate in acetone was determined. The viscosities of the nitrated cotton celluloses [1] were determined using 0.20% solution of the material in ethyl acetate. The intrinsic viscosities were calculated by the use of Hess and Philipoff's equation [7]. Degrees of polymerization were obtained from the intrinsic viscosities by Kramer's equation, using a constant of 230 for acetate in acetone and 175 for cotton and rayon in cupriethylene diamine [3].

Results and Discussion

Obviously, a number of kinds of nuclear radiation are available for experimental work of the type presently under consideration. Of interest here has been radiation from a reactor and from a Co^{60} course. In attempting to establish the order of stability of fibrous materials and/or the mechanism of degradation, it was important to know whether there was any essential difference in effect between these kinds of radiation and whether the ultimate effect might depend upon the rate of dose at which the fibrous substances were exposed. Early in this work, the use of fast neutrons was discontinued because very rapid changes were produced; reproducible effects could not be obtained from time to time. When the effect of thermal neutrons was compared with that of gamma rays alone at equal calculated dosages, the changes in the various materials appeared to be quite similar [12]. Moreover, there also appeared to be little effect of dose rate. These observations have now been quantitatively confirmed, and the appropriate data are presented below. In the discussion, for the sake of brevity, only illustrative data will be shown, but in every case they are typical of the results obtained throughout the work with all the fibrous materials that have been studied. Thus, where the effects of gamma rays from the Co^{60} source are shown, it may be assumed that thermal neutrons

had the same effect on the physical properties at the same total dose, and in each case the effect was not influenced by dose rate.

The data shown in Figure 1 are representative of the behavior of the materials when exposed at different dose rates to either thermal neutrons or to gamma rays. In this case the degree of polymerization of cotton as a function of total dose of radiation delivered is shown. Considering either type of radiation, it is clearly seen that the change in degree of polymerization is a function of total dose only and has no relation to the rate at which the dose is delivered. For example, the effect obtained after 256 hr. in the case of gamma rays at a low dose rate was identical when an equivalent amount of radiation was delivered in only 21 hr. Similarly, a 5-min. exposure to thermal neutrons at the fast rate was equivalent to about 70 hr. at the slow rate, and the change in degree of polymerization of the cotton was nearly identical. The data obtained with rayon and acetate were consistent with the findings on cotton. The same conclusion may be drawn with respect to the changes in the other physical properties of these fibers, such as those illustrated in Figures 3, 4, and 5.

In earlier work dealing with various elastomers and plastics, Bopp and Sisman [2] determined factors for the conversion of radiation dosage in terms of nvt (i.e., total thermal neutrons/cm.^{2}) to rads. Although there were some differences from polymer to polymer, the average figure was of the order of 1 nvt = 10^{-9} rad (or approximately 10^{-9} roentgen). When this was applied as the factor to equate the dosages delivered to the samples during exposure to thermal neutrons and to gamma rays, the data in Figure 2 were obtained. The neutron and gamma doses are both plotted in terms of roentgens against the degree of polymerization of cotton after exposure to each type of radiation. The curves very nearly correspond.[3] When tenacity was used as the criterion for degradation, identical curves were also obtained. Moreover, the changes in these properties produced in rayon and in acetate were also identical at equal calculated dosages. Thus, it appears that the conversion factor is applicable to cellulosic materials as well as to the synthetic polymers studied by Bopp and Sisman; the use of this factor permits the estimation of the effect, at a given total dose, of either type of radiation interchangeably.

Typical changes in tenacity, elongation, modulus, and stiffness for the cotton, rayon, and acetate yarns during exposure to gamma rays are shown respectively in Figures 3, 4, and 5. As already stated, when thermal neutrons were used as the radiation,

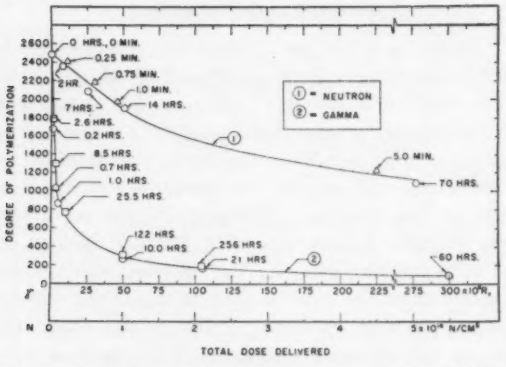

Fig. 1. Dependence of change in degree of polymerization of cotton on total dose of radiation.

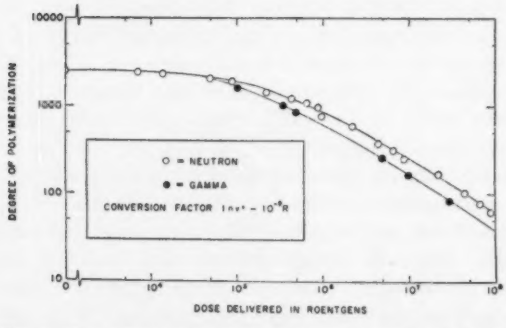

Fig. 2. Equivalence of gamma and thermal neutron radiation.

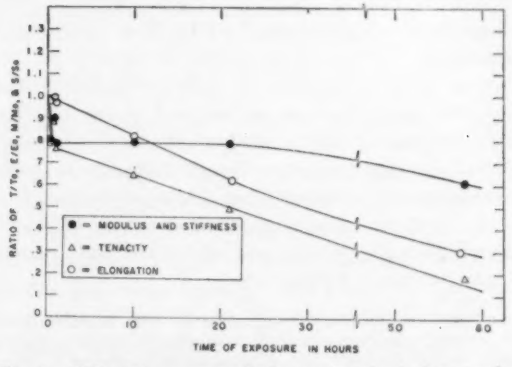

Fig. 3. Effect of gamma radiation on the physical properties of cotton; subscript $_0$ = untreated material.

[3] These data are not to be confused with Figure 1, where the total doses of the two kinds of radiation are not equilibrated.

almost identical changes in these physical properties were obtained at equivalent calculated dosages.[4]

The relationships between degree of polymerization (DP) and tenacity of the three fibrous materials after exposure for different periods of time to gamma rays are shown in Figure 6. By reference to Figure 1 it may be seen that relatively small doses of radiation caused a very rapid drop in the degree of polymerization of cotton. In Figure 6 it is shown that the tenacity decreases relatively little during the early period of exposure, and significant losses of strength are not obtained until the DP has dropped to the order of 1000–1200. This observation is consistent with the change in tenacity as a function of DP that is obtained when cotton cellulose is hydrolyzed by mineral acids. In the case of rayon and acetate, significant decreases in the DP are accompanied by significant losses in strength. This is also consistent with the general relationships found when these two materials are degraded by acid hydrolysis. From these data it would appear that the decreasing order of relative stability toward radiation of these fibers is acetate, rayon, and cotton.

In a previous paper [12] it was indicated that acid groups were formed in cotton and rayon when the fibers were exposed to either gamma rays or thermal neutrons. These groups would arise by oxidation. Thus, there may be some question whether the irradiated fibers might exhibit the property of alkali lability so often found in oxidized cellulose. If such were the case, the values for degree of polymerization that are presented might not be indicative of the chain scission produced by radiation alone, but would also include the effect of the alkaline cupriethylene diamine on the chain length of the irradiated sample. To elucidate this point, the irradiated cottons were nitrated [1] and the viscosities of the derived nitrocelluloses determined in ethyl acetate. The intrinsic viscosities of the nitrated samples were then plotted against the intrinsic viscosities obtained by the use of cupriethylene diamine. The straight line obtained

[4] During the course of this work a number of the exposures reported in this paper were repeated from time to time. It is worthy of note that good agreement relative to the changes in properties as a function of total dose of radiation was always obtained. The present data for changes in tenacity are also in good agreement with those reported by other workers in this field [5, 6].

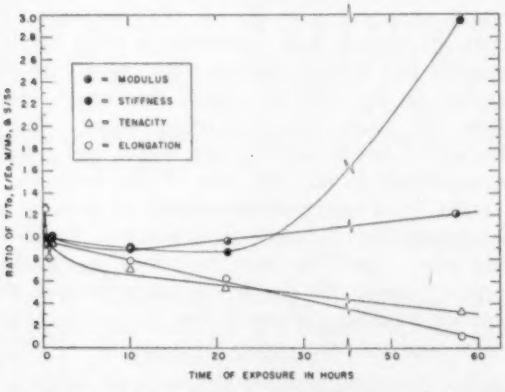

Fig. 4. Effect of gamma radiation on the physical properties of rayon; subscript $_o$ = untreated material.

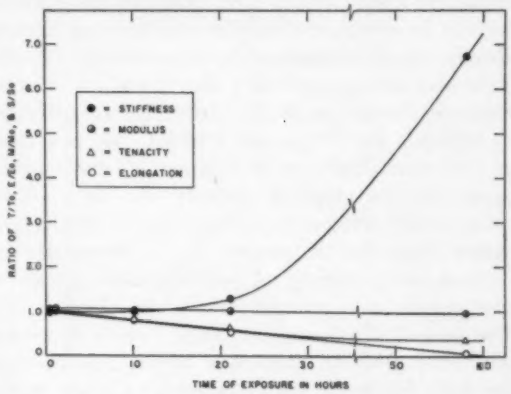

Fig. 5. Effect of gamma radiation on the physical properties of acetate; subscript $_o$ = untreated material.

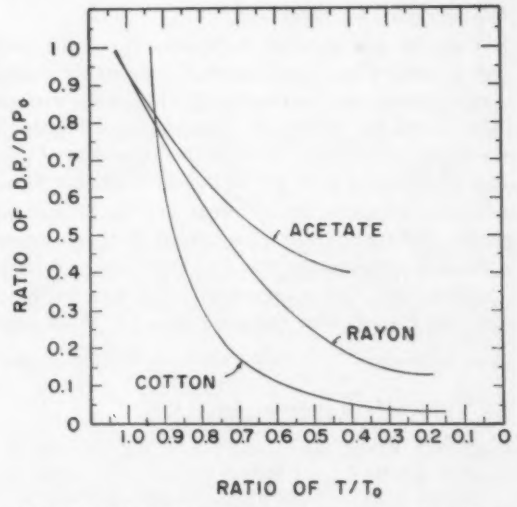

Fig. 6. Relationship between degree of polymerization and tenacity of irradiated cellulosic materials. Initial degree of polymerization (DP_o): Cotton, 2488; rayon, 298; acetate, 497. Initial tenacities (T_o): Cotton, 1.59 g./den.; H. T. rayon, 3.12 g./den.; acetate, 1.60 g./den.

is shown in Figure 7. The relation of strength to the intrinsic viscosities is shown in Figure 8. Thus, in spite of the presence of acid groups in these materials, indicated by dyeing experiments [12], there is no evidence of alkali lability; the chain lengths obtained by the viscosities in cupriethylene diamine apparently represent the effect of radiation alone.

Sippel [10], in studies of the effect of heat on fibers, derived a relation between fiber strength and the number of linkages broken by assuming that strength, as well as viscosity, is proportional to the number of unruptured cellulose chains in a cross section of fiber. Sippel later extended his work to include the effects of ultraviolet radiation and X-rays. Briefly, Sippel's treatment was as follows. In the molecular arrangement of a fiber there are N total monomer connecting links; of these, n are sensitive to radiation and m_0 are not affected. m_0 is a ratio of two constants,[5] one a function of stress–strain properties, the other of viscosity (or DP). This ratio can be determined directly from a plot of the natural logarithm of tenacity ($\ln T$) versus the reciprocal of the degree of polymerization. Such a plot results in a straight line whose slope is m_0. The numerical value of this slope (which is always positive [5]) gives the number of unruptured connecting links for every one ruptured by a given treatment; in Sippel's case, heat, ultraviolet radiation, and X-rays. He considered m_0 as a "fiber constant" characteristic of a treatment.

When the data obtained in the present investigation were examined by Sippel's method, interesting results were obtained, and the relative stability of the various fibers could be expressed numerically. Figure 9 shows the relationship between the logarithm of tenacity and reciprocal of DP of the three fibrous materials after exposure for different periods of time to gamma radiation. The comparison of these slopes in absolute terms means that for every linkage broken in acetate, 300 remain unbroken. It is interesting to note that Sippel, who regarded m_0 as a "fiber constant" for any given kind of treatment (radiation), obtained a value of 280 for acetate during exposure to heat or X-rays. The value of 300 found in this investigation is in good agreement with Sippel's work. The m_0 values for cotton and rayon, respectively, were 130 and 77. If it is assumed that the linkages broken are the glucosidic ones, or for that matter one linkage of any kind *per anhydroglucose unit* that will result in a shortening of the cellulose chain, a relative order of stability of the three fibers concerned may be obtained by dividing the initial DP by the m_0 values of the samples. When this is done, relatively more linkages are broken in cotton than in the other fibers, and the order of decreasing stability becomes acetate, rayon, and cotton. Relative stability (RS) numbers of about 2, 4, and 20 are obtained, respectively, as shown in Figure 9.

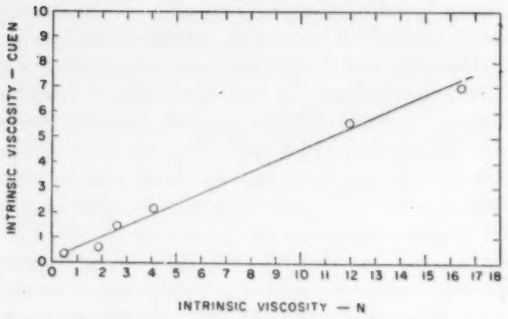

Fig. 7. Intrinsic viscosities of irradiated cotton in cupriethylene diamine and of nitrated cotton in ethyl acetate.

In the above treatment it was recognized that the determination of the relative orders of stability might be complicated by the fact that the acetate and rayon were in the form of filament yarn, while the cotton was in the form of staple. In previous work Orr et al. [8] have shown a relationship between strength retained and linkages broken when cotton was degraded by heat. It was found that cotton fibers gave one relationship and cotton yarns another. For a 10% loss in yarn strength, a 30% loss in fiber strength was found. In view of this behavior, a possible error in the relative orders of stability, as determined from Figure 9, was suggested. To clarify this point, the data were examined by another method. Using the degrees of polymerization, the number of linkages broken during the various treatments were calculated from the expression

$$L = 100\left(\frac{1}{\mathrm{DP}} - \frac{1}{\mathrm{DP}_0}\right)$$

[5] By definition

$$m_0 = \frac{K_1}{K_2} = \frac{\ln T_0 - \ln T}{\frac{1}{\mathrm{DP}} - \frac{1}{\mathrm{DP}_0}}$$

where

$$K_1 = \frac{\ln T_0 - \ln T}{t}$$

$$K_2 = \frac{\frac{1}{\mathrm{DP}} - \frac{1}{\mathrm{DP}_0}}{t}$$

and t = time of treatment.

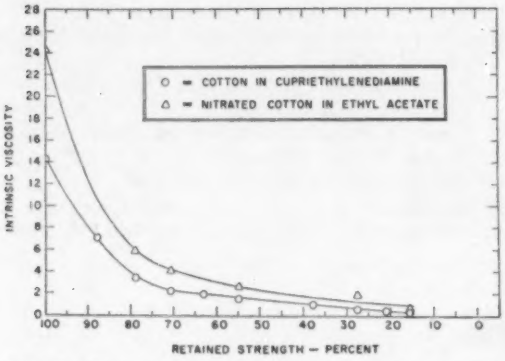

Fig. 8. Strength of irradiated cotton as a function of intrinsic viscosity.

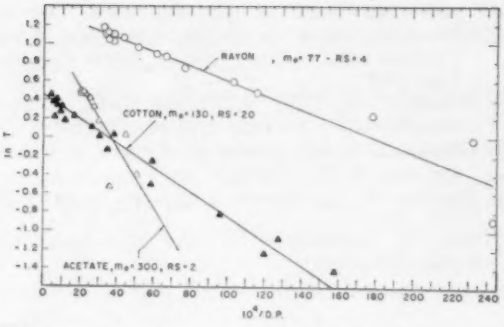

Fig. 9. Relative order of stability of cellulosic fibers according to the method of Sippel.

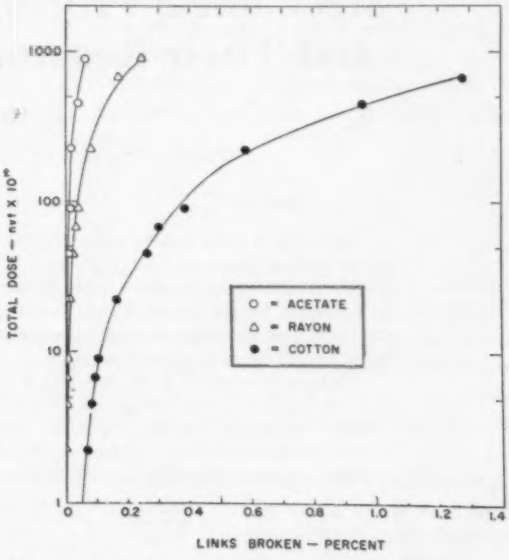

Fig. 10. Relation of linkages broken to total dose of radiation.

where L is the percent linkages broken, DP the degree of polymerization after treatment, and DP_0 the initial degree of polymerization [4]. The percent linkages broken by any given exposure was then plotted as a function of the total dose of radiation the sample received during that exposure. The series of curves shown in Figure 10 was obtained. It is clearly seen that at any given level of radiation the number of linkages broken in the materials increased in the order acetate, rayon, cotton. The orders of stability as obtained by Sippel's treatment are, therefore, confirmed.

It would appear from the above data that there are linkages in the native cellulose chain molecule susceptible to radiation that are not present in the regenerated materials. Considering the fact that the latter represent a native cellulose, or derivative, after being subjected to treatments that reduce the degree of polymerization, this observation is not surprising. Moreover, the concept of the presence of sensitive linkages in native cellulose already has been suggested elsewhere [9]. However, whether this behavior is associated with the presence of a peculiar type of linkage, or whether it is related to differences in degrees of crystallinity and/or orientation [11], is at the moment not clear.

Conclusions

The results of the present investigation have shown the following:

1. The degradation produced in cellulosic materials by nuclear radiation is a function of total dose and does not depend upon dose rate.

2. The effect on the physical properties of the fibrous materials of thermal neutrons was identical with the effect produced by gamma rays alone when the total doses of radiation were equated.

3. The relative order of stability toward nuclear radiation of the fibers considered in this investigation was acetate > rayon > cotton.

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Mechanical Properties of Cotton Fibers and Their Response to Mercerization[1]

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Abstract

Fifty fibers from twelve widely different cottons were mercerized by a single fiber mercerization treatment at 0.1 g. tension. The changes in the fiber extensibility properties brought about by this treatment were related to the degree of fibrillar orientation as estimated by the initial Young's modulus of elasticity. Although the fiber strength was increased by this treatment, no relationship was found between the magnitude of this increase and the degree of orientation.

Three of the cottons were mercerized at several tension levels ranging from no tension to 1.0 g. tension. The changes in fiber mechanical properties due to mercerization were found to be highly dependent upon the tension exerted on the fiber during the treatment. A tension of 0.1–0.2 g. appears to be the proper restraining force to overcome the tendency of cotton fibers to shrink during mercerization.

Introduction

Mercerization of cotton textiles to increase dye affinity, luster, and strength is a common industrial practice and can be accomplished by various combinations of temperature and sodium hydroxide concentration. Depending upon the tension exerted on the fiber during treatment, various effects such as change in crystalline modification of the cellulose (I to II), change in geometric shape and dimensions, change in fibrillar orientation, and change in mechanical properties of the fibers are observed. The changes in mechanical properties of the fibers are undoubtedly a manifestation of changes in crystalline structure and fibrillar orientation which are induced by the mercerization process. An excellent review of the effects of mercerization can be found in Ott and Spurlin [3]. Recent studies by Rånby and Mark [5] have indicated that mercerization takes place at characteristic NaOH concentrations for cellulosic materials of different origin. Thus, the characteristic concentration of NaOH for mercerization of wood pulps is 6–10%, whereas 8–11% is required for mercerization of cotton fibers. This difference may be ascribed to the varied internal order of these two cellulosic materials.

It has been thoroughly documented that the many varieties and strains of the world's cottons differ markedly in mechanical properties such as breaking

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strength, breaking elongation, and the Young's modulus of elasticity [8]. In many cases these differences can be ascribed to differences in geometrical factors of the fibers, most particularly the fiber cross-sectional area. However, even after normalization for fiber cross-sectional area, it is possible to discern differences in cotton fiber mechanical properties. These differences must then be ascribed to fundamental differences in the organization of the cellulosic material which accounts for roughly 95% of the cotton fiber. It has been shown that cotton fiber mechanical properties are related to the X-ray angle of cottons, which is a measure of fibrillar orientation [1, 7]. It is of interest to note here that extensibility properties of cotton fibers are highly correlated with fibrillar orientation, but that fiber strength is significantly correlated only at very short gauge lengths [6]. At gauge lengths higher than 5 mm. the determining factor that establishes cotton fiber strength is the occurrence and the magnitude of weak spots rather than fibrillar orientation [9].

The fact that cottons have been shown to have varying degrees of fibrillar orientation has led to the hypothesis that the different cottons should respond in a characteristic manner to a mercerization treatment. In the mercerization of yarns from widely different cottons it was found that these cottons differ in their response to the treatment [4]. This paper presents a report on the study of the response of several cottons to a single fiber laboratory mercerization treatment.

Experimental

Twelve cottons were considered in this research. The fiber mechanical properties indicate that these cottons cover a wide range, and on the basis of correlations shown previously between fiber elastic modulus and X-ray angle it is possible to conclude that these cottons represent a wide range in degree of fibrillar orientation.

The cotton samples were extracted in a Soxhlet apparatus with chloroform to remove the surface wax, in order to allow a more efficient mercerization. Fifty single fibers from each cotton were randomly selected and each fiber mounted on two brass tabs at an approximate gauge length of 0.5 in. The initial mechanical properties of the fibers were determined using the Instron Tensile Tester [10]. This was done in order to evaluate the fiber cross-sectional area by the vibroscope technique [2] and to determine the initial Young's modulus of elasticity. This latter value was used as an index of the fibrillar orientation of the cottons.

The 50 fibers, after a suitable relaxation period, were mercerized for 5 min. in 25% (by weight) NaOH at room temperature. The fibers were then washed in water, neutralized in 1% acetic acid, and again washed in water. After drying for 5 min. at 105° C., the fibers were examined on the Instron Tensile Tester in order to determine the mechanical properties of the cottons after the mercerization treatment.

No claim is made here that the mercerization conditions described above are optimum, nor that they represent a full duplication of commercial practice. It must be borne in mind that it was desired to ascertain the response of different cottons to an identical mercerization treatment rather than to optimize the several variables encountered during mercerization.

Discussion

All Cottons

The tension during mercerization that was chosen for the investigation of the response of the 12 cottons was 0.1 g. The order of magnitude chosen was dictated by the fact that this force is within the uncrimping region of the fiber; however, the actual value was chosen on the basis of experimental convenience.

Figures 1–3 show the changes in Hookean slope, breaking elongation, and breaking stress as a function of the initial Young's modulus of elasticity. Table I presents the experimental data, while Table II presents the correlation coefficients along with their statistical significance levels for the plots shown in Figures 1–3.

The change in Hookean slope is expressed as a percentage of the initial Hookean slope determined on each fiber prior to the mercerization treatment. Thus, in the case of this nondestructive property, it is possible to ascertain the change that each fiber undergoes during the treatment. The changes in breaking stress and elongation are also expressed as a percentage of the stress and elongation values of untreated fibers. Obviously, due to the destructive nature of the test, the initial breaking stress and elongation values had to be determined independently, on a separate set of fibers for each cotton.

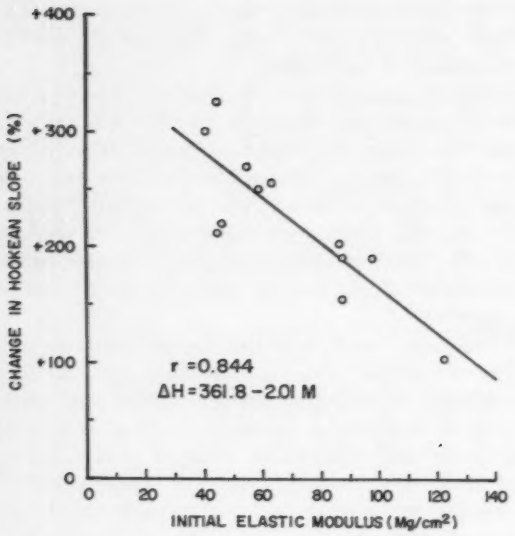

Fig. 1. Change in Hookean slope due to single fiber mercerization at 0.1 g. tension vs. the initial elastic modulus.

TABLE I. Changes in Fiber Mechanical Properties Due to Single Fiber Mercerization in 25% NaOH at Room Temperature. Tension = 0.1 g.

Cotton	Initial modulus of elasticity, Mg./cm.²	Change in fiber properties, %		
		Hookean slope	Breaking elongation	Breaking stress
Experimental strain	40.3	+300	−44.1	+27.2
Watson Mebane	44.4	212	40.6	17.1
Belgian Congo	44.5	326	38.2	73.6
Lengupa	46.0	220	40.3	1.5
Acala 4–42	54.2	270	29.1	29.9
Pima S-1	58.1	251	45.5	5.8
Deltapine 15	62.6	256	30.3	52.8
Sea Island	85.6	204	34.1	38.2
Karnak	86.9	155	34.2	0.7
Supima	87.2	191	21.5	30.2
Coastland	96.8	190	26.4	52.0
Triple Hybrid	121.5	105	5.3	35.0

TABLE II. Correlation Coefficients Between Initial Fiber Elastic Modulus and Changes in Fiber Mechanical Properties Due to Single Fiber Mercerization in 25% NaOH at Room Temperature. Tension = 0.1 g.

Fiber property	Correlation coefficient	Statistical significance level
Hookean slope	0.844	Better than 0.1%
Breaking elongation	0.835	Better than 0.1%
Breaking stress	0.061	No significance

Examination of Figure 1 shows that the change in Hookean slope is an inverse function of the initial elastic modulus. To the extent that the initial elastic modulus can be considered a measure of the degree of fibrillar orientation, it can be said that the change in Hookean slope due to mercerization is an inverse function of the fibrillar orientation [6]. Since the increase in Hookean slope is also a measure of the increase in fibrillar orientation, it appears reasonable that the cottons with an originally high degree of fibrillar orientation, brought about by a combination of genetic and environmental conditions during fiber growth, do not increase their orientation during mercerization as drastically as lower oriented cottons. The differences among the cottons as far as elastic modulus is concerned are greatly reduced, which indicates that the differences in degree of orientation are greatly reduced by the mercerization treatment.

Figure 2, which shows the change in fiber breaking elongation as a function of the initial elastic modulus, indicates the same sort of dependence of the manner in which the breaking elongation is al-

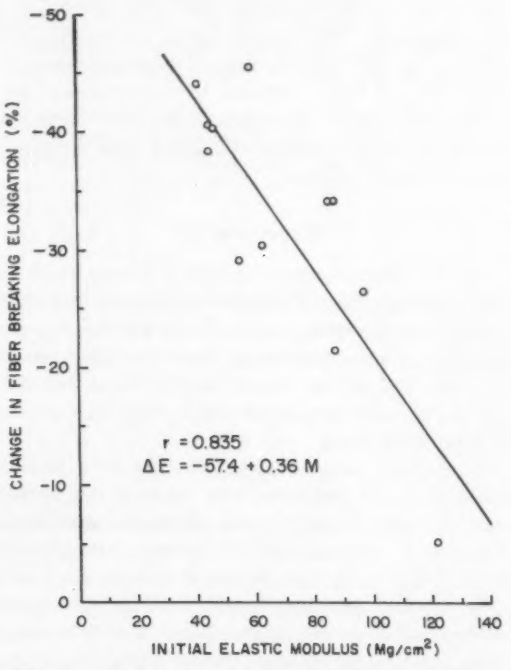

Fig. 2. Change in breaking elongation due to single fiber mercerization at 0.1 g. tension vs. the initial elastic modulus.

tered upon the initial modulus and hence upon the degree of fibrillar orientation. Cottons with a low degree of fibrillar orientation decrease their fiber breaking elongation much more during the mercerization treatment than fibers with an initially high degree of orientation.

It is indicated, therefore, that as far as fiber Hookean slope and elongation to break are concerned the response of the several cottons is related with a highly statistically significant coefficient of correlation to the degree of fibrillar orientation as estimated by the fiber elastic modulus.

As would be expected, the fiber breaking stress is also altered by the single fiber mercerization treatment. The change induced by the treatment varies considerably from no increase in breaking stress to an increase of 74%. While the increase in breaking stress varies over this wide range, it has not been possible to relate it to any known property of the cottons. As is shown in Figure 3 and presented in Tables I and II, no significant relationship exists between the increase in fiber breaking stress and the fiber initial elastic modulus. Thus, the increase in fiber breaking stress does not appear to be related to the degree of fibrillar orientation. This is not surprising since it has been shown that the absolute value of fiber breaking stress when measured at 0.5-in. gauge length is only weakly dependent upon the degree of fibrillar orientation, as indicated by the X-ray angle [6]. As far as fiber breaking stress at gauge length longer than 5.0 mm. is concerned, the dominant factor is the frequency and the nature of the weak spots along the fiber length [9]. It appears, then, that some of the weak spots found along the fiber's length are reparable by the mercerization treatment, while others are not reparable. It should not be expected that repair of weak spots be dependent upon fibrillar orientation of the major portion of the fiber. The complete lack of correlation between increase in fiber breaking stress and the initial modulus of elasticity (used as a measure of fibrillar orientation) therefore is not surprising.

Effect of Tension

The tension on the fiber during mercerization exerts a considerable influence on the manner in which the fibers respond to the mercerization treatment. Three of the cottons that were studied in this research were mercerized at several different tension levels ranging from no tension to 1.0 g. tension. The changes in mechanical properties of these three cottons at the several tension levels are

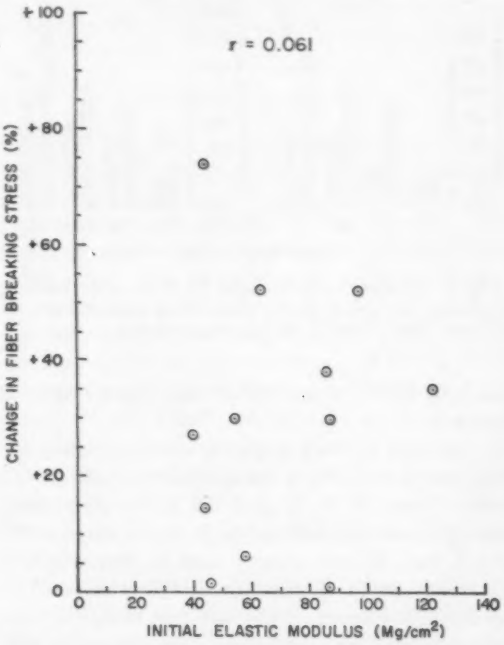

Fig. 3. Change in breaking stress due to single fiber mercerization at 0.1 g. tension vs. the initial elastic modulus.

TABLE III. Changes in Fiber Properties Due to Single Fiber Mercerization in 25% NaOH at Room Temperature at Several Tension Levels

Tension, g.	Change in Hookean slope, %			Change in breaking elongation, %			Change in breaking stress, %			Change in length, %		
	D	P	K	D	P	K	D	P	K	D	P	K
0	+17	−6	−9	+120	+100	+73	+12	−10	−10	−11.9	−14.9	−14.1
0.1	+256	+251	+155	−30	−46	−34	+53	+6	+1	0	+1.5	−0.8
0.2	+340	+288	+189	−39	−60	−54	+63	+19	+26	−0.7	0	+2.3
1.0	+526	+447	+385	−61	−74	−66	+87	+26	+40	+5.2	+7.5	+6.2

D = Deltapine 15; P = Pima S-1; K = Karnak.

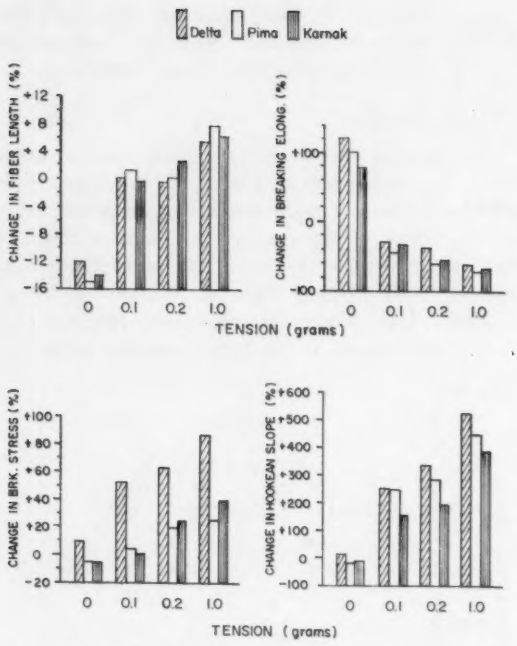

Fig. 4. Changes in fiber length, Hookean slope, breaking elongation, and stress due to single fiber mercerization vs. the tension on the fibers during mercerization.

shown in Table III and are presented graphically in Figure 4.

A decrease in fiber length is observed when the fibers are mercerized in the absence of tension. At tension levels of 0.1 g. and 0.2 g. the restraining force appears to be sufficient to overcome any shrinkage; in fact, in some cases a gain in fiber length is observed. At 1.0 g. tension there is a decided increase in fiber length. The fact that complete mercerization was attained during the treatment was established by examination of fiber cross sections.

The change in Hookean slope is also a function of the tension. With no tension there is virtually no change in Hookean slope. The increase in Hookean slope, when the fibers are under tension, increases with the tension, although not linearly.

The fiber breaking elongation increases markedly when the fibers are not held under tension during the treatment. In the presence of tension the fiber breaking elongation decreases. The magnitude of this decrease is a function of the tension, although a leveling-off value is being approached at 0.2 g. tension.

The fiber breaking stress is increased by mercerization under tension, but is somewhat decreased when mercerization takes place with no tension.

Conclusions

The manner in which cotton fiber extensibility properties are altered by mercerization is a function of the degree of fibrillar orientation of the cottons as measured by the initial modulus of elasticity. The decrease in fiber elongation to break and the increase in fiber Hookean slope are inverse functions of the fibrillar orientation.

The mercerization treatment increases the fiber breaking stress of most of the examined cottons, but this increase is not a function of fibrillar orientation. It is proposed that increase in breaking stress is caused by repair of weak spots along the fiber length and that this repair has no apparent relationship to the fibrillar orientation.

Acknowledgment

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An Automatic Vibroscope

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Abstract

A simple, automatic version of the vibroscope now frequently used for the determination of mass per unit length of fibers, filaments, and yarn is described. The automatic vibroscope gives continuous indication, is very fast in response, and permits the study of sorption kinetics in single fibers. It is also more sensitive than most manually adjusted versions. Details are given of the construction and operation of the instrument, and an assessment is made of the accuracy of measurement under usual operating conditions. Some other possible applications are briefly discussed.

Introduction

The vibroscope, introduced to textile science by Gonsalves [5] in 1947, has since been used extensively in the determination of mass per unit length of fibers, filaments, and yarns. It is based on the well known phenomenon of resonance in vibrating strings. If a string of length l and mass per unit length m is subjected to a tension T it may be resonated at its fundamental natural frequency f of transverse vibration, given by

$$f = \frac{1}{2l}\sqrt{\frac{T}{m}} \qquad (1)$$

(See, e.g., Wood [23].) If this resonant frequency can be determined and l and T are known, m can be calculated readily. Also, since in a uniform fiber or filament $m = \rho A$, where ρ = density of the fiber material and A = cross-sectional area of the fiber, if ρ is known A can be determined. This is the most common application [2, 9] of the vibroscope. Since m includes any sorbed water in the material, the vibroscope may also be used to measure regain, an application first pointed out by Sproule [12].

A number of methods [2, 9, 11] may be used to set the fiber (or filament or yarn) into transverse vibration; they include mechanical, acoustic, and electrostatic devices. Similarly, several ways of determining when the fiber is in resonance have been studied. These include direct visual observation using a microscope or projector, photo-electric, or mechano-electric methods.

A common operating procedure, after setting up the fiber at constant length and tension, is to adjust manually the frequency of excitation until the fundamental resonance is observed, as evidenced by a maximum in the amplitude of fiber vibration. The mass per unit length may then be determined from Equation 1. The precision with which the exciting frequency can be adjusted for maximum amplitude depends on the "sharpness of tuning" of the vibrating fiber system and the sensitivity of the amplitude sensing device. In certain cases small changes in mass per unit length are required to be measured, and the necessary high precision may be more conveniently obtained by use of a sensitive method suggested by Sproule [12], in which the phase shift in the fiber rather than the amplitude of vibration is observed.

The vibroscope in the simple form described is satisfactory for many purposes and is available commercially. There are applications, however, where it would be more convenient, or even essential, to have operation which is automatic in that the resonant frequency is indicated directly and continuously on some form of meter. This meter could be calibrated in mass per unit length, cross-sectional area, or fiber diameter. For instance, routine testing of fiber diameter would be accelerated, be less subject to error, and call for less skill on the part of the operator if no signal generator adjustment were required but the diameter simply read from a dial or counter. Again, in following the change in fiber regain consequent on a change in ambient relative humidity, the variations are frequently so rapid as

to preclude manual adjustment of the driving frequency to the resonant value.

To fill the need for automatic operation of the vibroscope in these applications and others, e.g., yarn evenness testing and hygrometry, development of an automatic vibroscope was undertaken. A previous paper [4] included a brief description of the instrument. The purpose of the present paper is to provide sufficient detail to allow the instrument to be built and operated.

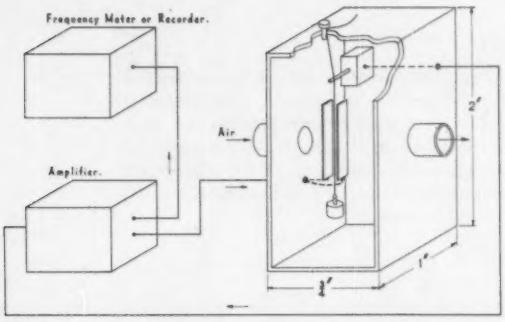

Fig. 1. Schematic arrangement of automatic vibroscope (not to scale). Fiber box shown with front cover removed.

Automatic Vibroscope Apparatus

The basic problem in the automatic vibroscope is to ensure that the frequency of excitation of the fiber is always equal to the instantaneous natural frequency. The most convenient way of doing this is to use the well known method of positive feedback (see, e.g., Parker [10]). Assuming the fiber to be initially in oscillation at its resonant frequency, its motion is detected by some form of transducer providing a corresponding electrical output, which is amplified. This amplified energy is converted back by a driving transducer into mechanical energy which is supplied to the fiber and just suffices to make good the losses of the vibration, which therefore proceeds continuously. The process is analogous to the methods in which a piezo-electric quartz crystal or a tun-

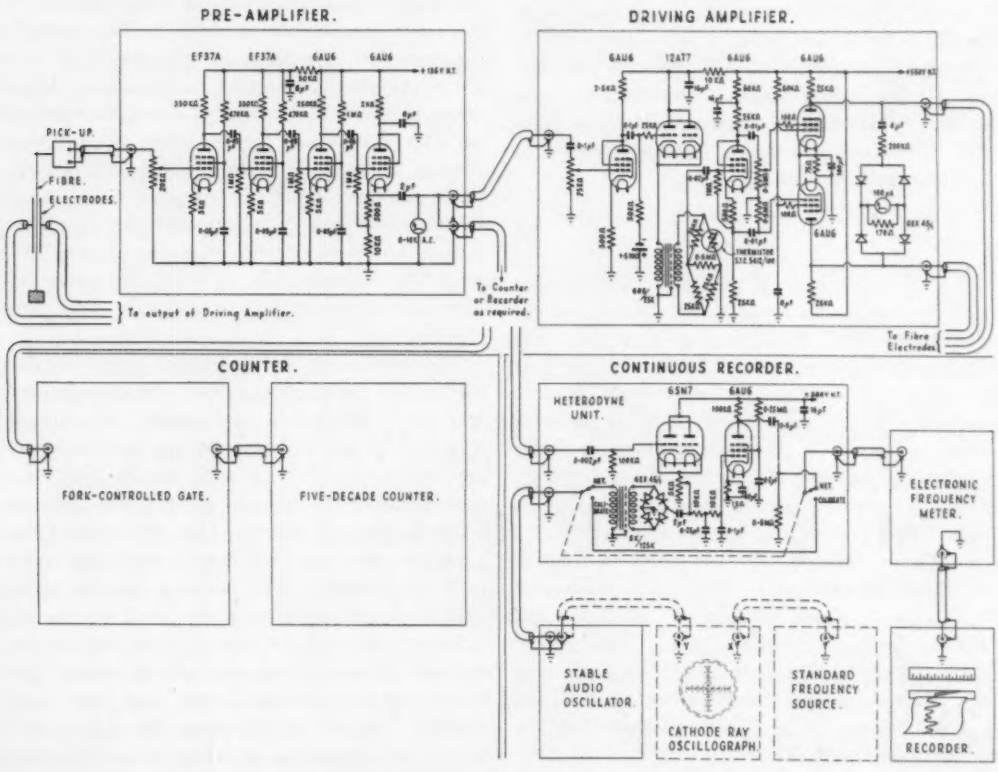

Fig. 2. Circuit diagram of amplifier and frequency measuring apparatus.

ing fork is maintained in oscillation to provide a reference frequency.

A form of the apparatus which we have found satisfactory for sorption studies and which could be used for diameter measurements is described below (Figure 1). The fiber is clamped at its upper end, passes over a smooth pin near that end so as to make an angle of contact with the pin of 5° or more, and is held in tension by a weight fastened to the lower end by cement or a clip. The pin is mounted in a standard piezo-electric pickup as used with long playing phonograph records. A very small fraction of the energy of transverse vibration of the fiber is abstracted by the pickup to produce an electrical signal which is normally about 5mV r.m.s. in amplitude. This is applied to an audio frequency voltage amplifier (see Figure 2) which is designed to produce, with this input, an output of at least 50V r.m.s. This output is applied across a pair of electrodes spaced about the fiber center. In addition, both electrodes are raised to a steady potential of 250V D.C. The latter causes a negative charge to be induced on the fiber at its center, and the action on this charge of the alternating field between the electrodes produces an oscillating mechanical force on the fiber. This force is in antiphase with the forces (principally air drag) opposing the vibration (a condition achieved by making the total phase change around the loop equal to zero at the natural frequency). The effect of this latter force is thus neutralized and the oscillation continues indefinitely. With values of fiber length and tension commonly used, the frequency of oscillation is in the range 400–3000 cycles/sec. and the amplitude of vibration of the midpoint of the fiber is 1–2 fiber diameters.

An important requirement of the detecting pickup is that it should not exhibit any mechanical resonances in or near the operating range of frequencies. Such resonances are characterized by large variations in phase shift between input and output of the transducer. Any phase change so introduced causes the oscillation frequency of the fiber to change until an equal and opposite phase shift occurs in the fiber, since it is a characteristic of any system in steady state oscillation that the phase shift around the oscillatory loop is always zero (see, e.g., M.I.T. Staff [13]). Thus, undesirably, the fiber is forced away from its natural frequency of vibration. Unfortunately, practical transducers do exhibit mechanical resonances, for example between the mass of the moving element and the compliance of its support, and it is necessary in practice to ensure that such resonances do not lie in or near the operating frequency range or, if this is unavoidable, that they are heavily damped to minimize their effect.

The electrostatic method of driving the fiber was suggested by Montgomery [9]. As well as having the desirable feature of providing a simple driving method without mechanical contact, it has two advantages for the automatic vibroscope. By obviating the need for a transducer having a moving element it eliminates troublesome mechanical coupling between driving and detecting transducers via a common mounting. This coupling frequently causes self-oscillation at some frequency quite unrelated to that of the fiber. Again, since there are no moving parts other than the fiber itself, the electrostatic drive is free from transducer resonances which, as we have already shown, can cause the frequency of oscillation to move away from the natural frequency of the fiber. (Photoelectric detection is similarly free from spurious mechanical resonances, so that a combination of this method of detection with electrostatic drive should be particularly advantageous.) The driving electrodes are of elongated form so that they act over the central two-thirds of the fiber length (see Figure 1). By this means the excitation in the fiber of odd harmonics of higher order than the fundamental is much reduced. (The even harmonics are not excited because of the longitudinally symmetrical driving arrangement.) Suppression of such harmonics minimizes the tendency of the vibroscope to oscillate at a frequency other than the fundamental and also improves the frequency stability (see, e.g., Terman [17]).

The amplifier is for the most part conventional in design, with negligible phase shift in the operating frequency range. An important feature is the inclusion of automatic gain control. The signal amplitude in an uncontrolled oscillatory loop will be limited by overloading of one or more stages, with consequent distortion. The resultant production of harmonics adversely affects the frequency stability. It is therefore desirable to stabilize the amplitude at a lower level; this may be done conveniently by introducing into the loop a Wheatstone bridge incorporating a thermistor [14]. In such a bridge, balance occurs at a particular value of voltage supply; over a range of voltages slightly less than this the bridge has the characteristic that its output falls steeply as the input is increased; hence control takes place in this region.

The amount of tension applied to the fiber should be high enough to straighten it but not so high as to cause significant longitudinal creep. These requirements are easily met in the case of uncrimped or slightly crimped fibers but not so easily in the case of finely crimped material; e.g., merino wool. In the latter case a compromise must be made; a typical value of tension on a merino fiber of 20μ diameter is 0.5 g., at which the crimp is practically removed; the corresponding strain is about 0.8%.

If when testing a number of fibers of different diameters the tension is adjusted in each case until the frequency is approximately the same, all fibers will be tested under the same stress, which is usually desirable. This follows from Equation 1, which may be rewritten

$$f = \frac{1}{2l}\sqrt{\frac{T}{m}} = \frac{1}{2l}\sqrt{\frac{T}{A\rho}} = \frac{1}{2l}\sqrt{\frac{S}{\rho}}$$

where A = cross-sectional area of fiber; ρ = density of fiber; and S = stress in fiber.

Setting-Up Procedure

In setting up the automatic vibroscope there is a procedure which is useful for obtaining the correct operating conditions. The oscillatory loop is opened at a suitable point, which is usually between two stages in the maintaining amplifier; a signal from an audio frequency generator is fed in at this point so as to cause the driving transducer to be energized. The ingoing signal and the signal returning from the other side of the opening in the loop are compared by connecting them respectively to the X and Y plates of a cathode ray oscillograph. Phase and amplitude relationships around the loop may then be studied by reference to the ellipse displayed on the oscillograph, a well known procedure in electronics [19]. After examination for absence of anomalous behavior of the phase and amplitude in the vicinity of fiber resonance, the phase shift should be adjusted to zero when the loop gain is a maximum; i.e., at resonance. The loop gain should then be adjusted to be slightly greater than unity. When these adjustments have been made and the loop closed again the fiber will oscillate continuously at its natural frequency, to the precision with which the latter can be determined.

Since the frequency of oscillation is always that for which the phase shift around the loop is zero, any change in phase shift which may occur in the maintaining circuit will automatically be compensated by a phase shift in the fiber. Because of the interdependence of fiber phase shift and frequency, a change in oscillation frequency away from the natural value will result. However, since the phase shift in the fiber varies rapidly with frequency in the neighborhood of resonance, such changes will usually be negligible.

Frequency Measuring Apparatus

The design of the frequency measuring apparatus (see Figure 2) depends on whether or not it is intended to observe rapid changes of frequency, and on the degree of resolution required. For example, in measurement of mass per unit length, where the result is effectively an average, high precision of frequency measurement may not be justified. In other cases, such as the accurate determination of fiber regain, the frequency measuring apparatus should be capable of measuring to better than the limit of stability of the vibroscope, which with temperature control to $\pm.5°$ C. and constant fiber parameters is one or two parts in one thousand of frequency over several hours.

When it is not required to measure rapid changes in frequency, a method [20] which has been found suitable is to use a four or five decade decimal counter to display the number of cycles occurring in a known time interval (e.g., 1 or 10 sec.), the latter being obtained from an electronic "gate" generator which is itself controlled by a standard frequency oscillator. When using the vibroscope for kinetic measurements of rapid sorption processes, the discontinuous nature of the above method of measuring frequency is inconvenient. A more suitable method is to use an electronic frequency meter [21] (such as are available commercially), in which the output voltage is proportional to the frequency of the applied signal and independent of its waveform and amplitude. The output is recorded by a graphic milliammeter or automatic potentiometer. The accuracy of measurement is greatly improved by heterodyning the vibroscope signal with an independent constant frequency, so that a small difference frequency results. The constant frequency is conveniently obtained from a stable audio oscillator which is periodically checked against a standard frequency source. The frequency difference is applied to the frequency meter and recorded. In this way frequency can be recorded to 1 cycle/sec. without difficulty. This arrangement of

the apparatus has been used successfully in studies of the kinetic sorption of water in wool fibers [4].

Accuracy

The accuracy achieved with the automatic vibroscope depends on what quantity (e.g., mass per unit length, regain) is being measured.[1] In measurements of mass per unit length the interest lies in the extent to which the value determined from application of Equation 1 differs from the true mass per unit length.

In the derivation of Equation 1 it is assumed that the string is uniform, perfectly flexible, and vibrating between fixed supports with infinitesimally small amplitude in a vacuum. None of these assumptions is strictly true, and mass per unit length calculated from Equation 1 will accordingly be in error; the errors are, however, usually small.

The effects of nonuniformity, stiffness, and the presence of air around the fiber have been examined by Montgomery and co-workers [8, 15, 22], who have applied to the vibroscope the fundamental investigations of Rayleigh, Seebeck, and Stokes. In practice the largest error will probably be that due to nonuniformity of the fiber, but its magnitude cannot be predicted without determining the nature of the nonuniformity. The effect is due to the fact that a variation in mass per unit length of an element of fiber changes the natural frequency by an amount depending on the position of the element, the amount being greatest for an element at the center and zero at the ends. Voong and Montgomery [22] have shown that in some 58's wool fibers the error averaged 3%; in coarse wool, 2%; and in 3-den. nylon monofilament, less than 1%. The error may be of either sign.

The effect of stiffness in the fiber is to raise its natural frequency [8]. The magnitude of the effect depends on the method by which the fiber ends are fixed; e.g., by clamping, passing around a pin, attaching to a weight etc.; it is greatest when both ends are clamped.

The presence of air around the fiber lowers its natural resonant frequency [15]. The effect is due to the addition of a mass of air to that of the fiber and to the viscous drag of the air.

The estimated magnitudes of the errors due to these effects are shown in Table I for the case of a wool fiber of 20μ diameter and 2.5 cm. length vibrating at a frequency of 2000 cycles/sec.

The effect of the ends not being rigidly fixed but capable of slight transverse deflection due to elasticity of the supports is to lower the natural frequency [7], but the effect will usually be negligible; see Table I.

The effect of finite amplitude of vibration [16] of the fiber is usually quite negligible provided the amplitude at the midpoint is not allowed to exceed a value of two or three fiber diameters.

When the vibroscope is used for relative rather than absolute mass per unit length measurements, as in regain measurements, the accuracy is substantially improved because the error terms discussed above cancel out to a large extent. For example, we may reasonably assume that the error due to nonuniformity is the same in the two measurements of mass per unit length required for regain determination, and similarly for the error due to movement of the supports. The errors due to stiffness will not wholly cancel out because of changes in Young's modulus and fiber diameter with regain; similarly, the errors due to the effect of air around the fiber do not wholly cancel out because of diameter changes. The magnitude of the errors arising from the two last-mentioned effects are shown in Table I for the case of the wool fiber already quoted; it is seen that in this case the effects balance each other out.

Finally, there is a correction to be applied in regain measurement due to longitudinal swelling of the fiber. This arises from the fact that the resonant frequency varies as $1/(\text{length})^{\frac{1}{2}}$, as may be seen by rewriting Equation 1.

$$f = \frac{1}{2l}\sqrt{\frac{T}{m}} = \frac{1}{2}\sqrt{\frac{T}{Ml}}$$

where $M = ml$ = total mass of fiber between supports. For example, the longitudinal swelling of a wool fiber is about 1% between 0 and 15% regain; hence the correction at the latter value will be -0.5% of regain.

Further Applications

We have so far discussed the use of the automatic vibroscope for the determination of the mass per unit length of fibers and yarns and for studies, both equilibrium and kinetic, of the amount of water sorbed from the vapor phase by hydrophilic fibers (use of

[1] It is assumed that the phase shift in the vibroscope circuit has been adjusted in the way described earlier, so that the fiber is oscillating at its natural frequency.

TABLE I. Errors in Determination From Equation 1 of Mass Per Unit Length and Regain, Calculated for the Case of a Wool Fiber of 20μ Diameter

Length = 2.5 cm., Oscillation Frequency = 2000 cycles/sec.

	Mass per unit length		Regain		
Source of error	Magnitude of error	Remarks	Magnitude of error	Remarks	Reference
Nonuniformity	Up to 3%	See text	Negligible	See text	Voong and Montgomery [22]
Stiffness	−1%	Both ends of of fiber clamped	0.2% of regain	At regain of 15%*; both ends of fiber clamped	Montgomery [8]
Surrounding air	1.5%		−0.2% of regain	At regain of 15%*	Stauff and Montgomery [15]
Motion of supports	0.3%	One end of fiber fixed, other attached to slightly yielding support (in this case, needle of conventional phonograph pickup)	Negligible	See text	Rayleigh [7]
Finite amplitude of oscillation	Negligible	See text	Negligible	See text	Stuart [16]
Length swelling			0.5% of regain	At regain of 15%*	

* This is the approximate regain of wool at 65% R.H., 20° C.

the instrument can, of course, be extended to vapors other than water vapor). The automatic vibroscope may also form the basis of a hygrometer with several desirable characteristics. If a suitable hydrophilic fiber having no sorption hysteresis is used there will be a single-valued relation between its mass per unit length and the ambient relative humidity, so that the vibroscope may be calibrated in terms of the latter quantity. The instrument would be direct reading, relatively fast in response, and would make a very small demand on the moisture content of the surrounding air. The type of fiber used should be selected to display no anomalous (long term) sorption effects [1, 6].

A further application of the automatic vibroscope lies in the measurement of variation in mass per unit length along a yarn. The yarn is run past a measuring head where a short section of it is automatically set in vibration at its resonant frequency. This instrument has been described in detail by Downes and Leary [3].

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Reaction Spinning of Fibers

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Abstract

A method of forming fibers which is relatively new, yet of potentially wide application, is described herein. The process may be termed "reaction spinning." In reaction spinning, the usual viscosity increase required between spinneret and windup is accomplished by chemical reaction. Incompletely polymerized or incompletely solubilized polymer is forced through an extrusion die or spinneret under such conditions of rate, temperature, concentration, and activation that the polymerization proceeds rapidly within the fiber after it leaves the spinneret. This causes the soft extruded fiber to set to a solid or suitably semisolid state in fiber form on the windup package, where further reaction will complete the transformation to the desired fiber properties. Principles and experimental examples of the process are described.

MAN HAS devised many ways, some quite ingenious, of forming fibers. Yet the silkworm and the spider have a method which man seems not to have used as fully as he might. As the animal ejects prepolymer from its spinnerets it is mixed with a catalyst causing very rapid further polymerization. The drawing action of the animal, besides helping to pull out the material, also orients and crystallizes the fibers during the spinning [4]. Their process may be regarded as a type of "reaction spinning." The process of reaction spinning brings about the usual viscosity increase required between spinneret and windup by means of chemical reaction within the fiber. Broadly speaking, the process consists of forcing incompletely polymerized or incompletely solubilized polymer (or polymers) through an extrusion die or spinneret under such conditions of rate, temperature, concentration, and activation that an important part of the polymerization proceeds rapidly within the fiber after it leaves the spinneret. This causes the soft, extruded fiber to set to a solid or semisolid state in fiber form on the windup package. On the windup package, further reaction will complete the full transformation to the desired fiber properties. The process, in its simplest form, will be in one stage, but obviously may be multistaged if desired.

The three most commonly used methods of forming fibers involve the extrusion of a viscous liquid through spinnerets. The methods differ principally in their manner of producing hardening of the extruded fiber. One, called "wet spinning," uses a second liquid to precipitate and harden the extruded solution of polymer after it has left the spinneret. Chemical reaction between the two solutions is usually involved. Another method, "dry spinning," uses the evaporative loss of solvent to harden the extruded solution of polymer. The third method, "melt spinning," uses the cooling of the melt to harden the extruded, heat-stable polymer. Viscose

rayon fibers, for example, are made by wet spinning, cellulose acetate fibers by dry spinning, and both nylon and glass fibers by melt spinning.

Another method is gaining in use for the spinning of polymers for which no acceptable solvent is known (e.g., polytetrafluoroethylene). It consists of pumping a suitably viscous suspension of the polymer in liquid through a spinneret, then fusing the residue by heat to form the finished continuous fiber [1]. The method is quite like that used in Italy several centuries ago for spinning ceramic fibers from an extruded suspension of clays in a "milk." Heat was applied to set the fiber form. Similar practice is also said to have been used for the forming of precious metal fibers.

Fibers are frequently formed without the aid of spinnerets. Fibers may be drawn from a molten pool of viscous polymer, as for example, in the case of rockwool and spun-candy fibers. Analogous procedures are also carried out using the analogues of wet or dry spinning. There is no *a priori* reason why it should not be done with reaction spinning. In fact, in one experimental example cited below (phenol-formaldehyde), just such a means of forming fibers was accomplished.

Fibers may, of course, be machined from bulk polymer, much as is steel wool. Fibers may also be cast. Short fibers may be made by the "fibrillation" of bulk polymer by using a mechanical action combining rolling and shearing (e.g., wood linters, or as in the familiar act of erasing with a rubber eraser). In each method, the possibility of using reaction spinning techniques continuously is apparent.

One of the more interesting methods of fiber forming is that from surface layers. E. K. Rideal in 1948 described a process whereby British workers found that under certain conditions a monolayer of polymer (e.g., protein) spread on a water substrate may be compressed laterally and forced to roll up into a fiber. Continuous filaments may be so formed by pulling at right angles to the direction of compression. The fiber then consists of a bundle of partly parallelized chain molecules lying in a spiral blanket-like roll. That this particular method will ever be commercially useful is questionable, but the uniqueness of the method and product is noteworthy. A related method, interfacial spinning, consists of pulling a fiber continuously from the interface of two liquids, one a prepolymer or polymer solution, the other a reactant or preciptant causing the fiber to set.

Aside from the operations of spiders, silkworms, and the like, one of the earliest known deliberate examples of reaction spinning was made at the Naval Research Laboratory in 1942. Fibers were reaction spun there by the writer using a silicone oil and an acid catalyst.

Silicone-Oil Fibers

Silicone oil was brought to a near-gel by the action of heat and oxygen. Nordhausen sulfuric acid was then added in about 1% volume to the viscous oil and the mixture rapidly extruded through a single hole spinneret. The material extruded as a fiber, which hardened considerably on standing. After 5 min., the fibers were hard and white, though not particularly strong (about 0.1 g./den. tenacity and 3% break elongation). Better properties doubtlessly could be developed by further experimentation. Some work has been done abroad in a related field —one that might be termed "reaction casting." There, polymer that solidifies rapidly because of inherent reaction is continuously cast. This experience doubtlessly will be of value in developing continuous reaction spinning [6].

An instance may be cited of reaction spinning by control of the change of state [3]. An ammoniacal solution of a cellulosic polymer was mixed with the stoichiometrical equivalent of strong acid solution containing a large amount of dissolved polyamide (protein). The mixture rapidly gelled, for the solvent power for the dissolved polymers was greatly diminished. The rapidly setting material may be extruded through a spinneret to form a fiber and then washed free of the salt.

Further very recent work on reaction spinning at the Plastics Laboratory at Princeton University has shown that the principle is capable of broad application. The following examples show that the principle may be applied to several types of polymeric reactions.

Phenol-Formaldehyde Fibers (No Catalyst Present)

An alcoholic solution of phenol containing a stoichiometric excess of formaldehyde was evaporated by warming to drive out the alcohol and the contained water. Continued heating caused the mass to become tacky to the point that fibers could be drawn from the melt. The withdrawn fibers were then

passed, while under mild tension, through a hot air chamber at about 140–200° C. The fibers then "froze" by internal cross-linking reaction in about 1 min. to become solid at that temperature and were removed in the fiber form.

Polyacrylic Acid and Polystyrene Fibers (Peroxide Catalyzed)

Inhibitor-free acrylic acid or styrene, to which 2% by weight of benzoyl peroxide was added as catalyst, was heated to initiate polymerization. When the mass became quite tacky, it was quickly chilled and broken up. Portions were transferred to an already hot (100° C.) extruder provided with a single spinneret hole, and the melt was rapidly extruded as a fiber. The extruding fiber was passed slowly (0.1 ft./min.) through a hot (140–200° C.) air chamber during which time further polymerization proceeded. Upon leaving the hot air chamber, the fiber was chilled and examined.

Polyacrylic Acid Fibers (Peroxide and Irradiation Catalyzed)

Inhibitor-free acrylic acid, to which 2% by weight of benzoyl peroxide was added, was heated to induce polymerization. When the mass had become quite tacky, it was rapidly chilled, broken as above, and transferred to the spinning machine. Here it was heated for about 1 min., then extruded in fiber form slowly. The extruded fiber was drawn slowly (0.1 ft./min.) under an intense ultraviolet lamp for a distance of about 6 in. to further polymerize it while in the fiber form. The fibers were then collected.

Cross-Linked Polyurethane Fibers

To 20 ml. of hexamethylene diisocyanate was added 15 ml. of tetramethylene glycol. The mixture was then heated to about 150° C., and 3 ml. of 1-2-6-hexanetriol was added as a cross-linking agent. The mixture was then heated and stirred to further the polymerization. After 10 min., the melt could be spun into weak (ca. 0.1 g./den.) filaments. These filaments were then passed through a hot air chamber (temperature about 180–250° C.) for between 30 and 60 sec. The emerging cold-drawable fibers were stronger (ca. 1–2 g./den.); see apparatus in Figure 1.

In this example, a difficulty of reaction spinning with potentially highly cross-linked polymers became apparent. Its nature and the means of overcoming it will be of interest for other cases. If the reacting polymer stays too long in the extruder or in the prefiber form, the reaction may easily proceed too far. This may make it difficult or impossible to form fibers in the existing equipment. In the above cross-linked polyurethane, this required termination of the run to clean up the extruder. Cleanup was accomplished most simply by adding a chain-cutting reactant to the overdeveloped melt—in this case by adding *m*-cresol. It was also found possible, but less convenient, to use overheating to degrade the polymer and so to reliquify it. From this experience one may anticipate the necessity for a method of continuous control of the melt viscosity during the reaction spinning of cross-linked polymers. The viscosity may be raised, lowered, or held sensibly constant by the desired monitored input ratios of cross-linking, linear-linking, and chain-cutting reactants, during long-term continuous reaction spinning. Doubtlessly there is some such mechanism in spiders and silkworms to relieve related constipation in their extrusion systems.

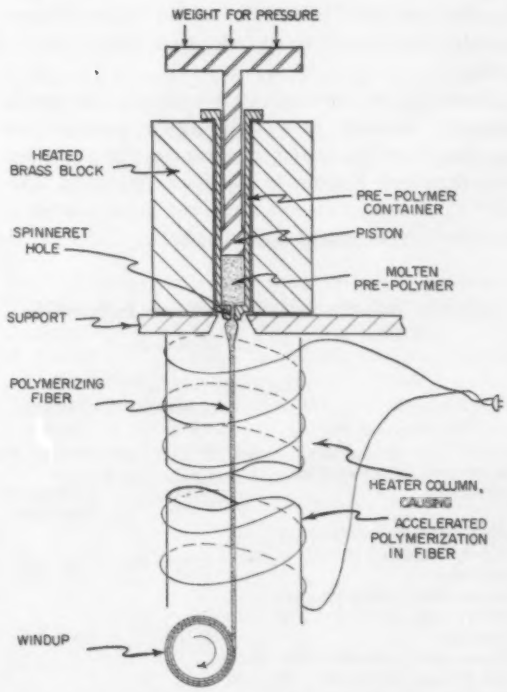

Fig. 1. Schematic diagram of reaction spinning.

Polyurethane Fibers

Tetramethylene glycol (0.0618 *M*) was heated in a water bath to 100° C. To the hot liquid was added 0.0620 *M* of hexamethylene diisocyanate. The temperature rose quickly to 145° C., and the viscosity increased considerably in the next 8 min. until the sample was chilled rapidly by pouring it out on an aluminum block. The brittle mass was crushed to a powder and quickly transferred to a small hot extruder. The extruder was held at 172° C. by thermostatically controlled heating. After a 7.5-min. holding period in the extruder, the polymer melt viscosity rose to a point where filaments could be extruded at 90 psi. The extruded filaments passed down into an 18-in. long heated air chamber below the spinneret. The air temperature was not uniform in the chamber, but was between 200° and 250° C. for the top 13 in. of the length. During their 1.5-min. run through this hot zone the filaments polymerized, as evidenced by their considerable increase in strength and molecular weight. These fibers could easily be cold drawn to strong fibers (about 4 g./den.), whereas those which were extruded from the spinneret either before or after the period the heated air chamber was used could not be cold drawn without trouble; then they formed only weak fibers (ca. 0.5 g./den.).

The molecular weight, as evidenced by the specific viscosity, increased in a fairly regular manner during the time the polymer spent in the extruder. Post-extrusion heating of the fibers in air at 200–250° C., however, caused a marked improvement of the fiber properties, as Table I shows.

TABLE I. Reaction Spinning of a Linear Polyurethane

Polymer condition	Specific viscosity at 0.375% wt./vol. in *m*-cresol at 25.0° C.	Tenacity, g./den.
Prepolymer, before entering extruder	0.33	too weak to measure
Polymer fibers, extruded after 7.5–8.5 min. at 172° C. in extruder	0.63	ca. 0.5
Polymer fibers, extruded after 10–11 min. at 172° C. in extruder	0.77	ca. 0.5
Polymer fibers, extruded after 8.5–10 min. in extruder and dwelling 1.5 min. in 200–250° C. air chamber	1.08	ca. 4.0

It is of interest to ask how much reaction must take place in the fiber during the interval between extrusion and windup to cause a change in viscosity sufficient to allow a fiber to be laid down. The viscosity change must be sufficient to permit the fibers to retain their shape sensibly, so that subsequent reaction in the windup package can bring about the ultimate desired degree of fiber hardening. Tackiness, causing the fibers to adhere to each other and to the windup machinery, can be controlled in most cases by the use of a surface lubricant, either liquid or powder. No precise figure can now be set for the viscosity which a freshly spun fiber must have to retain its shape for a brief span on the windup package, while the viscosity is there undergoing still further increase. One may, for the sake of giving an order of magnitude to the results, assume that a viscosity of 1,000,000 poises is desired upon the arrival of the fiber at the package. With the above assumption of 1,000,000 poises for the viscosity upon arrival of the fresh fiber and 10,000 poises for the viscosity upon extrusion, one may proceed to estimate for a typical polyester what degree of reaction is indicated.

Using the data of Flory [2] for the polyester $-O(CH_2)_{10}OCO(CH_2)_6CO-_m$, one may calculate that a viscosity of 10,000 poises at 200° C. occurs when p, the extent of reaction, equals 0.825. Similarly, a viscosity of 1,000,000 corresponds to $p = 0.895$. Hence, an increase in the extent of reaction of 7% would raise the melt viscosity from melt-extrudable to fiber-packaging viscosity. This rather small degree of reaction to produce high degrees of viscosity change is typical of high polymer systems. In fact, such a degree of change (7%) is larger than required for many systems (*viz.* the silicone oil–Nordhausen sulfuric acid system).

The timewise change of viscosity in high polymer systems generally follows an exponential course, as indicated in Figure 2. Let us consider the case in which only chemical reaction produces viscosity changes in the fiber between the time it leaves the spinneret (or prepolymer pool) and arrives at the windup as a hardening fiber, i.e., pure reaction spinning. As observed earlier, about 7% or less of the total polymerization reaction must take place in that interval. For example, in reaction spinning fibers at 20 m./min., with a spinneret-to-windup distance of 2 m., a time of 0.1 min. would be permitted for the viscosity-raising reaction to take place in the

fiber. This indicates roughly that one would choose to operate with reaction systems in which the time to go from near-monomer state to near-completion of the polymerization is several minutes or less. Such rapid polymerizations are not uncommon, but their control is bound to be critical. The slower the reaction to be used on a particular reaction spinning operation, the easier will be the control problem, but the slower will be the rate of production. Good instrumentation will be essential in minimizing polymer losses and in obtaining optimum operating speeds.

The term "reaction spinning" is capable of very broad application. It may be said to be taking place wherever there is any reaction at all which raises the polymer viscosity or molecular weight after the fiber shape is formed. In this sense, the examples cited earlier are indeed all examples of at least partial reaction spinning. In terms of this very encompassing definition, it is probable that reaction spinning takes place in many instances not normally considered as reaction spinning—for example, during the melt spinning of polyamides in dry gas. In the latter case, the principal mode of bringing about the increase of fiber viscosity is by the chilling of the melt. Reaction spinning is usually quite secondary in importance in such a case. On the other hand, reaction spinning is clearly the major mechanism for producing high viscosity and molecular weight in the first two and last two examples.

As may be seen from the data available, the field of reaction spinning has been only lightly explored. Its full potential is, it would seem, large and rather promising. Of the various chemical systems which one might survey for use in this matter, those involving the rapid vinyl type polymerizations seem most feasible. Some very rapid polycondensation reactions are, however, available, such as in the cases of the polyglycolethers, the N-carbo-anhydrides, and various gelling or cross-linking reactions using powerful reagents (e.g. Nordhausen sulfuric acid). Reaction spun fibers of mixed polymers, using mutual chemical precipitation to achieve rapid coagulation and viscosity increase, also offer considerable scope for fruitful study. The production of novel fibers of high insolubility or high temperature stability may well appear in the future to be best accomplished by means of reaction spinning.

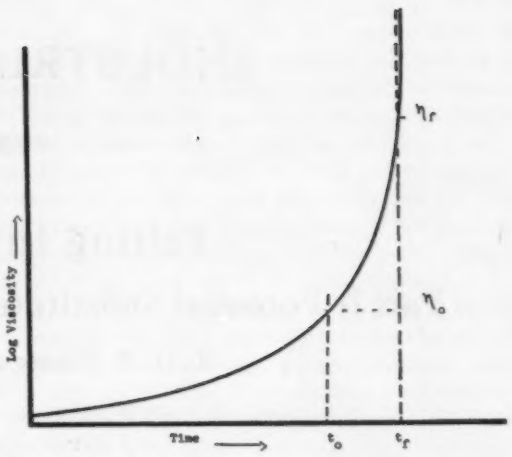

Fig. 2. Viscosity-time requirements of polymeric material for reaction spinning. η_o, t_o = viscosity and time at point of leaving spinneret; η_f, t_f = viscosity and time at point of solidification into fiber.

Acknowledgment

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INDUSTRIAL SECTION

Felting Investigations

Part I: Potential Substitutes for Rabbit Fur in Hat Felts

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Abstract

Recent uncertainties in the supply of rabbit fur for felt hat manufacture have prompted inquiries regarding the use of other fibers for fur felts, and the felting characteristics and histological structures of a number of possible alternatives have therefore been investigated. It is concluded that opossum, fox, and wallaby fur might be blended in considerable proportions with rabbit fur without seriously affecting the quality of the felt, and that crimpless (mutant) Merino wool might be used in small proportions.

Introduction

There is considerable interest in this laboratory in the process of felting, not only because of its central importance in the manufacture of hat felts, floor felts, and other types of felt, but also because of its relevance to the finishing of many types of woven fabric and to the development of processes for conferring shrink resistance. In the present series of papers it is planned to report a number of investigations of the felting and shrinkage processes. The present study was prompted by inquiries from fur felt hat manufacturers regarding possible alternatives to rabbit fur, as the supply of this material has been seriously curtailed following the introduction of the myxoma virus into the rabbit population of Australia.

The animal fiber in most abundant supply is wool, and it is extensively used for milliners' felts, but at present men's hats produced from wool are considered inferior to those produced from rabbit fur. Although the felting properties of rabbit fur have been studied in some detail [2], the reasons for its superiority over wool for this particular purpose have not been established in detail; in the present investigation some major differences between the two types of fiber are noted. However, in comparing the quality of hats produced from rabbit fur and from wool it must be borne in mind that the methods of hood manufacture are entirely different and that this may contribute to the difference in quality. In the fur process the skin is carrotted with a solution of mercuric nitrate in nitric acid to enhance felting, and the cut fur is carried in a stream of air onto a perforated metal former. After treatment with hot water the hood is detached and milled. Wool, in contrast, is not carrotted but is wound onto a former as a loose carded web in readiness for milling.

An important consideration in searching for a substitute for rabbit fur is that existing fur machinery should require as little modification as possible. The type of wool used for hat felt does not appear to be suitable for felting on the standard rabbit fur felt equipment, so a number of other animal fibers are also considered.

Materials and Methods

The fibers examined in the present investigation are tabulated below. The names enclosed in brackets are those used in the text.

1. Merino wool, 64/70's quality A.W.R.C. Type 61, cleaned by extraction with organic solvents and water (Normal Merino wool).

2. Merino wool, 64/70's quality, which had been specially selected for wool felt hat manufacture. The sample, which had been commercially scoured and carbonized, was neutralized and washed in water before use (Hatting wool).

3. Wool with no crimp, obtained from a mutant Merino sheep (Mutant Merino wool).

4. Rabbit (*Oryctolagus caniculus*) fur from skins prepared for use in the hat industry (Rabbit fur).

5. Kangaroo (*Macropus* sp.) fur (Kangaroo fur).

6. Bennett's wallaby (*Wallabia ruficollis*) fur from large and small skins (Wallaby fur, large and small).

7. Fox (*Vulpes vulpes*) fur (Fox fur).

8. Brush tail opossum (*Trichosurus vulpecula*) fur (Opossum fur).

9. Ardil, 3½ den. of staple length ¾–1 in. (Ardil).

Felting characteristics were assessed by means of the apparatus shown in Figure 1, which consisted of a pair of flat rubber-covered plates, 15 × 15 cm., between which the sample was milled. A reciprocating action with a stroke of 10 cm. was used at the rate of 60 cycles/min.; the load could be varied between 0.5 and 4.5 kg. In an attempt to simulate the manufacturing processes used in fur felt production, 5 g. of fibers was teased onto a 16-mesh brass wire sieve, 10 × 10 cm., and consolidated by spraying with water at about 80° C. for 20 sec. The mat of fiber thus obtained was rolled up in a piece of cotton cloth and milled for 10 sec., after which time it was sufficiently firm for the area to be measured. The milling was then continued and the felt removed at intervals for measurement of the area shrinkage. The felt was rotated through 90° after each measurement so as to retain an approximately square shape.

The fox, kangaroo, wallaby, and opossum fur samples were prepared by trimming the protruding portions of the guard hairs from the pelts and carrotting with mercuric nitrate. This solution was prepared by dissolving one part by weight of mercury in four parts by weight of concentrated nitric acid (sp. gr. 1.38–1.40). After the reaction had ceased the solution was heated until clear and then diluted to a specific gravity of 1.06. The carrotting liquid was brushed over the fur, and the pelt was dried overnight at approximately 45° C. After being cut, the carrotted fur was stored for a minimum period of three months prior to the felting tests to simulate the industrial carrotting process. Where necessary, the wool and the fur samples were cut to approximately the same length as the commercial sample of rabbit fur, thus minimizing any differences in felting characteristics due to varying fiber length.

Fur structure was investigated by cutting out a small piece of skin about 1 × 3 mm. and mounting it between lantern slide cover glasses. When the slide was placed in a photographic enlarger the shadows could be recorded directly on bromide paper. Staple structures in wool and Ardil were recorded in the same way. Cross sections were cut using a Hardy microtome, and their profiles were revealed by mounting whole fibers in Sirax, a high refractive index mountant ($n = 1.8$). Scale patterns were obtained by means of celluloid casts [5] formed on cover slips which were inverted on the microscope stage and photographed unmounted.

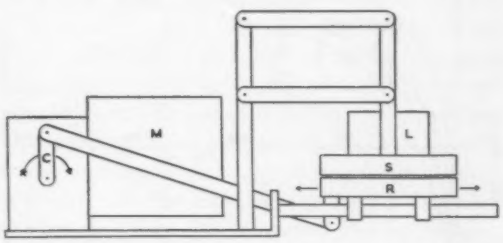

Fig. 1. Apparatus used to determine felting characteristics. The felting action was obtained by rubbing between a stationary plate (S) carrying a load (L) and a reciprocating plate (R). The reciprocating action was obtained by means of an electric motor (M) which operated a crank arm (C) through a reduction gear.

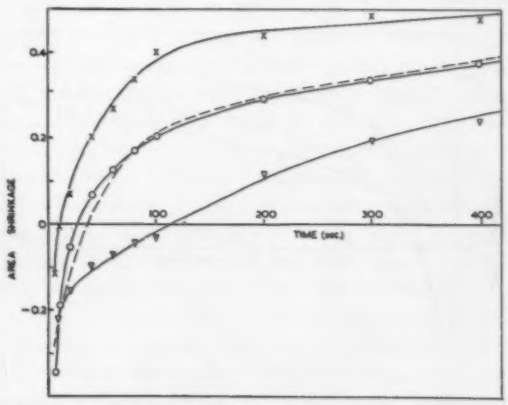

Fig. 2. The dependence of area shrinkage on time of felting in rabbit fur (- - -), Merino wool (▽), hatting wool (○) and mutant Merino wool (×). Area shrinkage was defined as: initial area (100 cm.²) − area/initial area.

Results

Preliminary tests showed that a suitable rate of felting was obtained in the milling apparatus with a load of 1.5 kg.; this was used throughout. In replicate experiments on an individual fiber type it was found that the area shrinkage, defined as (initial area − area)/initial area, at a given time was not reproducible. Further investigation showed that this was due to a variable spreading of the fiber mat when tipped from the sieve, which gave a variable value for the initial area. It was found, however, that if the initial area was taken as that *before* tipping from the sieve (100 cm.²) the area shrinkage/time curves were extremely reproducible apart from the first few points.

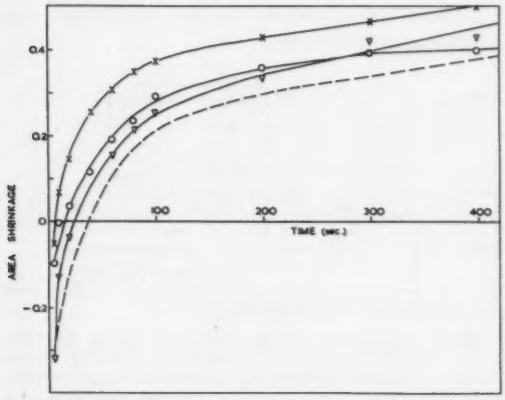

Fig. 3. Area shrinkage vs. time curves for kangaroo (×), fox (▽), and opossum fur (○) compared with that of rabbit fur (- - -).

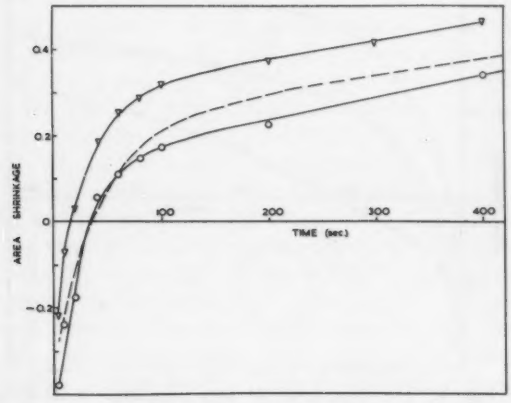

Fig. 4. Area shrinkage vs. time curves for two samples of wallaby fur, (○) large, (△) small, compared with that of rabbit fur (- - -).

The results of a representative series of measurements are presented in Figures 2, 3, and 4, in which

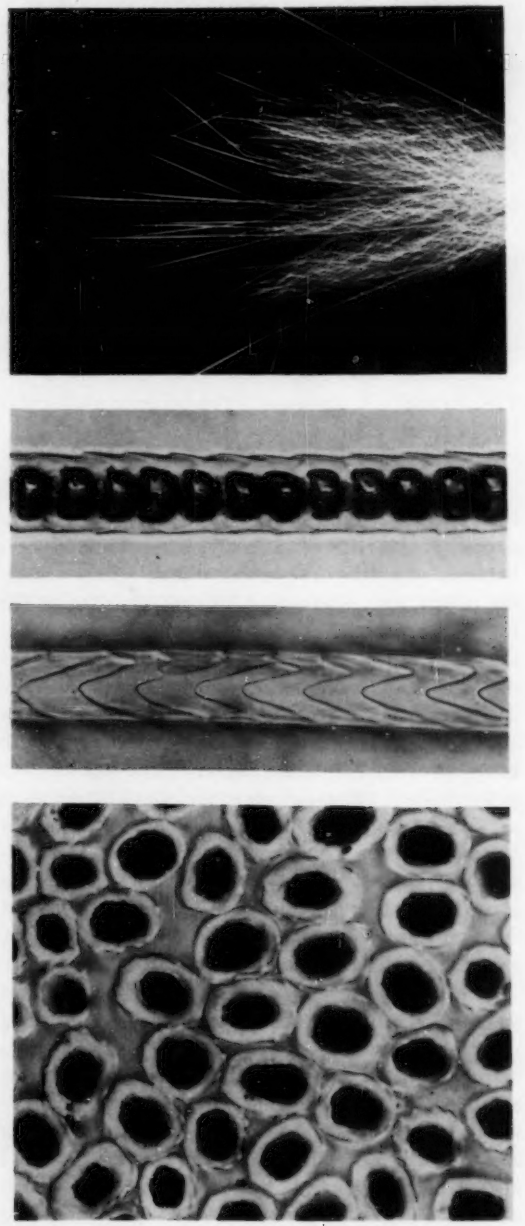

Fig. 5. Photographs illustrating the main structural features of rabbit fur likely to contribute to its felting properties. Top to bottom: fur structure (× 2.5); profile and medulla (× 750); scale pattern (× 750); cross section (× 750).

area shrinkage, defined now as (100 − area in cm.²)/100, is plotted against time. The negative values near zero time merely reflect the extent of spreading when the fiber matt was tipped from the sieve.

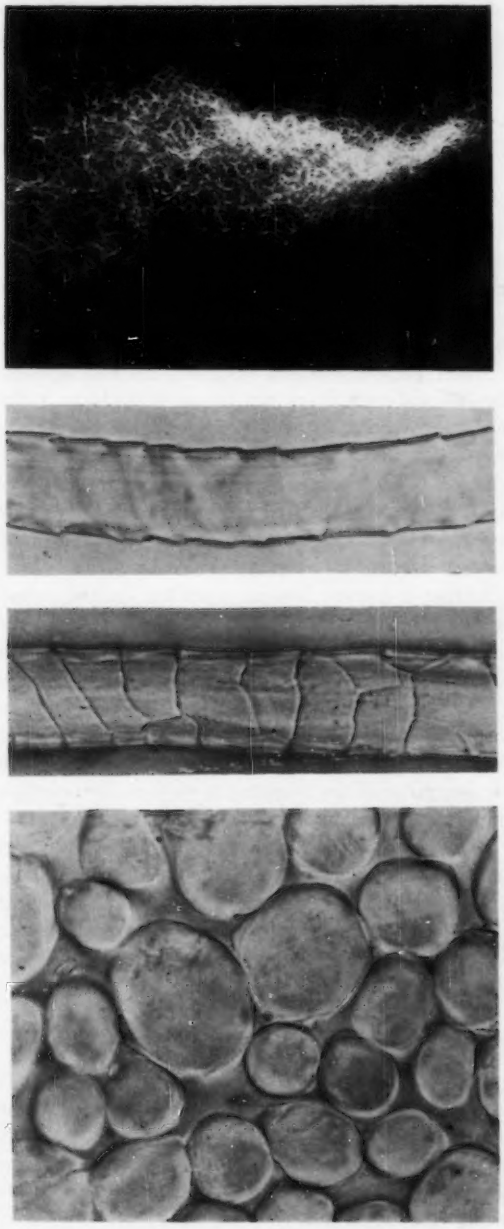

Fig. 6. Hatting wool; details as in Figure 5.

The main structural features of rabbit fur, hatting wool, kangaroo fur, fox fur, opossum fur, and Ardil are illustrated in Figures 5–10. Apart from difference in crimp, the other wools used in the present investigation were similar as regards scale pattern, profile, and diameter to the sample illustrated in Figure 6.

Discussion

It will be seen from Figures 2–4 that all the felts examined showed a rapid reduction in area during the first 100 sec. followed by a much slower rate of shrinkage which was approximately constant between 100 and 400 sec. For convenience these will be referred to as the first and second stages of felting.

Rabbit Fur and Wool

It is clear from Figure 2 that rabbit fur and normal Merino wool have quite different felting characteristics. The initial shrinkage of the fur was much greater than that of the wool, while in the second stage the rate of shrinkage of the fur was much less. The mutant Merino and hatting wools showed a variable amount of shrinkage during the first stage, but in both cases the shrinkage rate during the second stage was less than that of normal Merino wool.

It is interesting to note that the curve for hatting wool followed that of rabbit fur very closely.

The main structural difference between normal Merino wool and the other wools examined was in the nature of the crimp. In the hatting wool the crimp was not well marked, while in the mutant Merino wool it was absent. The fact that normal Merino wool exhibited the least shrinkage and mutant Merino the greatest shrinkage during the first stage of felting strongly suggests that crimp plays an important part in determining the felting characteristics of a fiber. Although crimp has been considered in previous work [3] as a factor influencing felting, little emphasis has been placed on it. However, preliminary reports of current investigations by Veldsman [4] appear to support our conclusion.

Crimp has another important bearing on the potentialities of wool as a substitute or diluent for rabbit fur. As mentioned earlier, the fur fibers are carried in a stream of air onto a perforated metal frame during the manufacturing process; entanglements between groups of crimped fibers would be a distinct disadvantage at this stage. Of the wools examined

so far, that of the mutant Merino is the only one to which this objection does not apply.

Although attempts to blend wool with rabbit fur have not been successful in the past, it is possible that mutant Merino wool, which is a furlike fiber, may prove to be more suitable. Attempts to blend in a high proportion of mutant Merino wool would, however, be expected to lead to a deterioration in felt quality. This may be appreciated from a consideration of certain histological features of the two fibers shown in Figures 5 and 6. The average diameter of the mutant Merino wool ($\sim 20\mu$) is significantly greater than that of the fur fibers ($\sim 15\mu$), so that a somewhat harsher hand is to be expected. The flexural rigidity, which plays an important part in determining hand, depends upon the fourth power of the diameter, so that a considerable difference in hand is to be expected from quite small differences in diameter. The effect of the medulla in rabbit fur in reducing the flexural rigidity would be only slight, as the main contribution comes from the outer regions of the fiber.

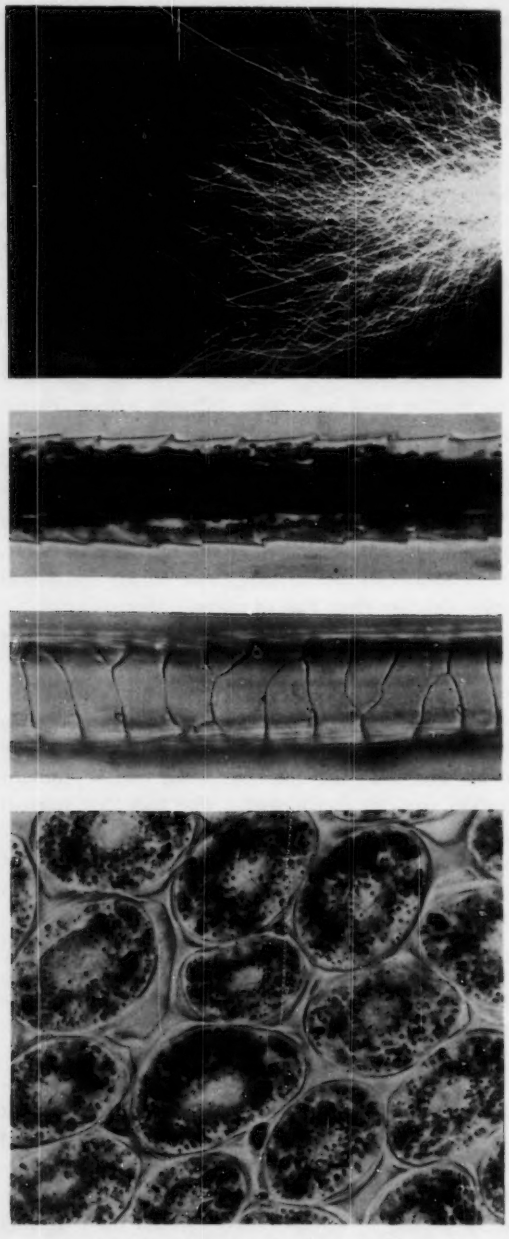

Fig. 7. Kangaroo fur; details as in Figure 5.

Although the medulla does not materially affect the flexural rigidity of fur fibers, it leads to a significant reduction in weight per unit length as compared with a nonmedullated fiber of the same diameter. The introduction of a high proportion of mutant Merino wool as a diluent would therefore be expected to lead to both a deterioration in hand and an increase in weight.

Although mutant Merino wool has limited potentialities as a fur diluent, its possible value in the production of wool felts should not be overlooked. The felting characteristics of hatter's wool are seen from Figure 2 to be intermediate between those of normal Merino wool and mutant Merino wool; this suggests that the latter wools might be blended in varying proportions to give any desired intermediate felting characteristics.

In a practical trial at a hat factory 20% of mutant Merino wool was successfully incorporated with rabbit fur in a felt hat. Although this product was not very different from the pure rabbit fur product, those experienced in the hat industry were able to detect slight deficiencies.

Other Animal Fibers

Felts can be made from many animal fibers; it was suggested to the authors that kangaroo fur might be suitable for the production of fur-felt hats. This suggestion is particularly attractive, as the kangaroo is a serious pest in certain parts of Australia. Following the same line of reasoning, wallaby, fox, and opossum fur were also investigated.

The main structural features of kangaroo, fox, and opossum fur are illustrated in the photomicrographs of Figures 7, 8, and 9. Kangaroo fur contains a proportion of guard hairs, but the main bulk consists of elliptical medullated fibers about $20 \times 30\mu$ in cross section; that is, considerably coarser than rabbit fur. The scale pattern is very similar to that of wool, but crimp is almost entirely absent.

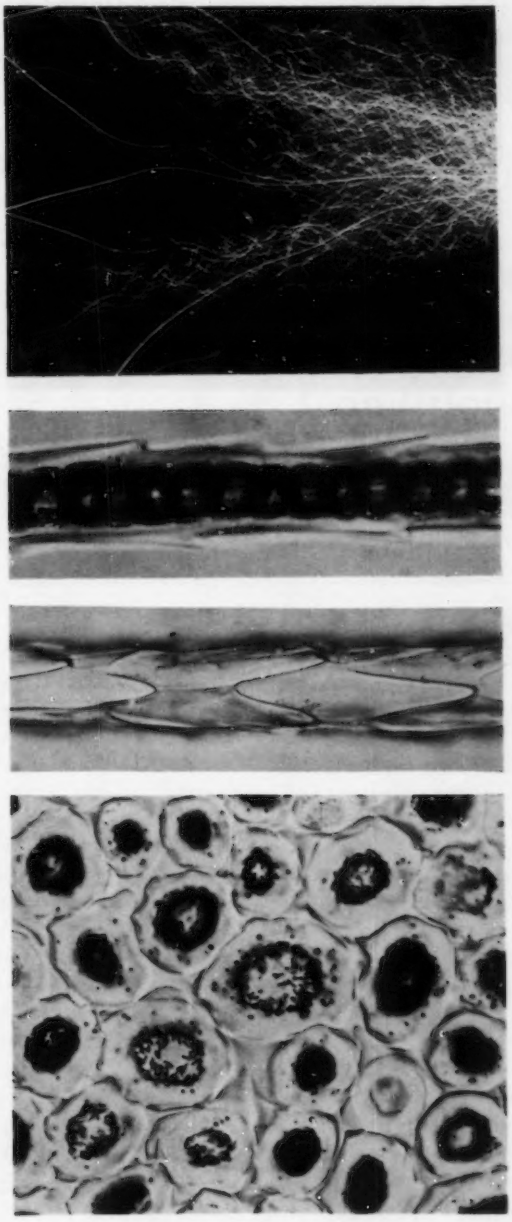

Fig. 8. Fox fur; details as in Figure 5.

Fox and opossum fur are very similar to rabbit fur in many respects. In both cases the medulla was of the uniserial ladder type; the mean diameters were about 20μ in fox fur and 16μ in opossum fur. The scale patterns, however, differed from that of the rabbit, being classed as diamond petal in both cases [5]. Also, the profiles show that the scale tips protruded to a much greater extent.

All the fur fibers mentioned could be felted satisfactorily after carrotting; the felting characteristics are shown in Figures 3 and 4. In every case the felting curves were sufficiently similar to that of rabbit fur to suggest that they could be used as alternatives.

Of the animal fibers investigated, opossum fur therefore appears to be the most closely related histologically to rabbit fur, while at the same time the felting characteristics are very similar. The average fiber length in the opossum pelt used in this investigation was somewhat greater than is general in rabbit fur, but this probably could be reduced during processing if necessary.

Synthetic and Regenerated Fibers

While animal fibers such as opossum fur could be used for fur-felts either alone or blended with rabbit fur, synthetic and regenerated fibers may be used only as diluents, as they do not possess a directional friction effect. The microscopical structure of a $3\frac{1}{2}$ den. regenerated protein fiber (Ardil) which has been used for this purpose is illustrated in Figure 10; it can be seen that, apart from minor cracks and fissures, both surface and profile are smooth. Although as much as 20% of this type of fiber has been successfully incorporated into rabbit fur felts without serious loss of quality [1], considerable difficulty has been experienced in industry in obtaining uniform blending and dyeing. The same difficulty is to be expected with fibers such as Fibrolane, Merinova, and Zycon; we have not considered these fibers further, as they are clearly of very limited value.

Conclusion

In the present investigation attention has been concentrated on felting rate and histological structure, and, while these properties serve to indicate

possible rabbit fur diluents, many other factors must also be taken into account, such as feel, tightness, pliability, and mellowness.

Bearing in mind the desirability of retaining existing machinery for fur felt production as nearly as possible in its present form, the most promising substitute among the fibers examined appears to be opossum fur.

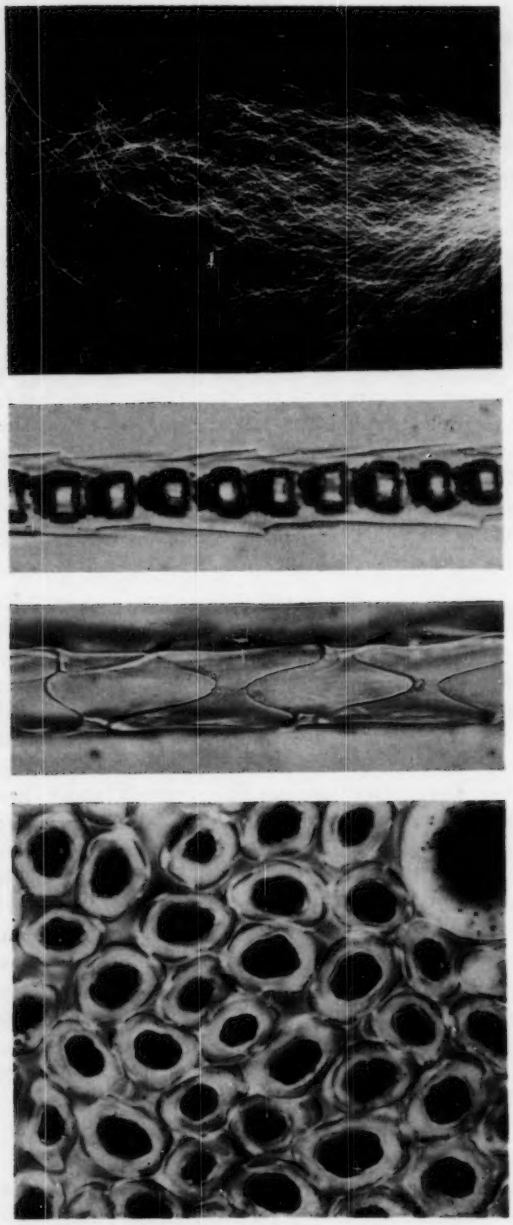

Fig. 9. Opossum fur; details as in Figure 5.

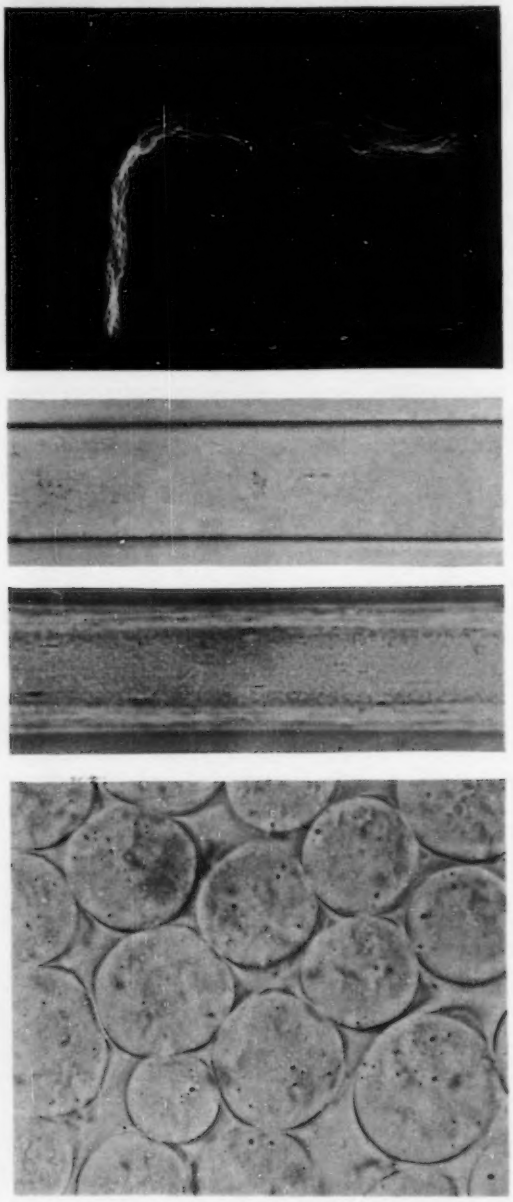

Fig. 10. Ardil; details as in Figure 5.

It is unlikely, however, that the supply of rabbit fur will fall sufficiently for a substitute to be considered; the trend is more likely to be towards the use of blends. The results presented here suggest that opossum fur, fox fur, and wallaby fur could be incorporated in considerable proportions, and mutant Merino wool in a somewhat smaller proportion, without significant reduction in felt quality.

Acknowledgments

We are indebted to United Felt Hats Pty. Ltd., Melbourne, for the supply of rabbit fur and hatting wools; to Dunkerley Hat Mills Ltd., Sydney; and to Dr. B. F. Short of the C.S.I.R.O. Sheep Biology Laboratory, for samples of mutant Merino wool.

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Color Change in Raw Cotton Related to Conditions of Storage

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Introduction

Since color is one of the measurable grade factors on which the quality of raw cotton is judged, there is considerable interest in knowing the degree of color change associated with different conditions of storage. Samples classed White when put into storage may be classed Spotted when withdrawn, or cottons put into storage as Spotted may come out as Tinged. For the general case, such facts are recognized [4, 5], but for specific cases, especially when the financial consideration involved may be large, the question recurs regarding the extent to which this reflects a real change.

Standards for grade will themselves change, a fact recognized for many years as a serious problem in standardization and reported elsewhere [6, 7]. But very little has been reported that is based on storage under a planned series of known conditions of humidity and temperature. Enough has been done to indicate that a combination of high temperature and high humidity will cause more color change in a given period than a combination of high temperature and low humidity, and that both combinations will cause more change than those involving moderate temperatures and humidities. Cottons held under refrigeration seem to show little or no color change. But no information has been available for specifying optimum conditions of temperature and humidity under which there would be a minimum of cotton color change. If it were financially feasible to meet such conditions, it would be a matter of very practical importance to all who now store cottons. If it should prove difficult or expensive to meet such specifications, this information would still be of great practical value even if its use were limited to storage of cotton standards after their adoption, for it would save a great deal of time, money, and argument relating to their continued validity and use.

At the 1956 Universal Cotton Grade Standards Conference it was pointed out that studies made on returned standards showed that color deteriorates more rapidly in use than had been supposed. This lowering of color through use (the samples get dusty and therefore measure darker), plus the yellowing that occurs during normal conditions of storage, all pointed toward the desirability of establishing a validity period on standards at 12 months. Universal grade standards conferences usually are held every

three years. Since preliminary results of laboratory studies indicated that cottons held under refrigeration changed very little in comparison to changes in cotton held at other more usual conditions of room storage, it seemed feasible to pass extra boxes at the 1956 conference and place enough of them in refrigeration to issue a new set annually to each signatory between conferences. While preliminary refrigeration studies were not carried out under controlled conditions, results seemed so favorable that at the 1956 conference storage of 1956 standards was proposed, and it was promised that during the three years before the next scheduled conference (1959) a controlled experiment would be made.

The study now reported was initiated immediately following this 1956 conference, and while it will be continued for the promised three-year period (so that the extent of any further change in that time may be known), results for the first year have proved so decisive and so useful that they are reported, for they seem to provide sufficient basis for setting specifications of temperature and humidity for holding cotton in storage to prevent or retard its color change.

Conditions of Storage

Through Dr. H. C. Diehl, Director of the Refrigeration Research Foundation, a series of storage chambers were located at Beltsville, Maryland and Experiment, Georgia, in which other tests were being processed. Through the cooperation of Dr. Harold T. Cook, Biological Sciences Branch of the Agricultural Marketing Service at Beltsville, and Dr. J. G. Woodroof of the Food Processing Division of the Georgia Experiment Station at Experiment, Georgia, space was made available in these chambers to store sets of cotton samples for periods up to three years under the following conditions:

Temperature, F.	Relative humidity, %
* 0°	H
22°	H
32°	H
38°	H
40°	H
45°	H
50°	H, 85%
50°	L, 50%
60°	H
70°	L, 50%
* 70°	H, 85%–90%
85°	H
*100°	H, 90%
*100°	L, 50%

Asterisks indicate storage at Experiment, Georgia; the other conditions are those used at Beltsville, Maryland. Except as noted humidity was uncontrolled and is marked H (high); the L (low) humidity in each case was held at 50%. In most chambers fruits and vegetables were being tested; some were even rotting, so that storage conditions were not always entirely suitable for cotton. However, use was made of what was available.

Material Stored and Method of Color Description

There are 24 grades of upland cotton now in effect [1]. Of these, 11 of the White and Tinged grades are represented in physical form by a 12-sample official box and a 6-sample guide box. In addition, 4 Spotted grades are represented in physical form by 6-sample field trial boxes. Following the 1956 conference, sufficient sets of these 15 grades were set aside to use in this study.

Samples from 6 bales are used in each White grade, 1 sample from each in the 6-sample box and 2 in the 12-sample box. The bales in each grade come from at least four different cotton growing areas: bales 1 and 2 come from the South Central area, bale 3 from the Southeast, bale 4 from the West, and bales 5 and 6 from the Southwest. In the Spotted and Tinged grades there may be no more than 3 bales used, these being selected to represent the color range of the box from the whitest to the yellowest samples.

Figure 1 is a diagram (based on the 1953 cotton grade standards) [6] used on the Nickerson-Hunter Cotton Colorimeter on which the measurements reported from this test were made. To understand this diagram one should know that while color has three dimensions (hue, lightness, and chroma), the hue for cotton is so nearly constant that measurements of lightness and chroma are sufficient to define the color of cotton grades, a fact that is convenient, since it allows use of the two-dimensional diagram shown. The Hunter scales used in this instrument indicate in a vertical direction the percent reflectance (R_d), which is a measure of the lightness of a sample, and in a horizontal direction Hunter's $+b$ which, for this instrument, indicates the degree of yellowness (with hue constant) and thus provides a measure of chroma. High grades are toward the top of the diagram, low grades toward the bottom; gray colors are toward the left, and yellow tinged or

stained colors are toward the right. The original of this diagram is made in a size that fits over the diagram on the instrument, so that indicated points may be plotted directly.

On this diagram the color range of each of the bales used in the 1956 standards and Spotted boxes is shown in the odd-shaped numbered enclosures. As may be seen, there is some overlapping of color in the Good Middling bales into Strict Middling (for it was not possible in all cases in 1956 to obtain bales to represent a full color step above Strict Middling), but otherwise there is no overlapping. The number in each enclosure indicates the bale position in the box, 1 to 6.

Samples in the test were individually measured for color at the beginning of the storage period and each time they were withdrawn, but in Figure 1 the range is shown for each bale. This range may represent measurements on as many as 1000 or more samples from each bale.

Color After Storage

So many samples are involved in each set that in order to show the overall picture, results are averaged for each grade and are shown on diagrams in Figures 2–12. Identification of the scales on the diagrams is omitted to save space; they are identical to those in Figure 1, which is reproduced in a large enough size to provide details. The small circle at the center of each grade represents its average color, and differences that develop during the various stor-

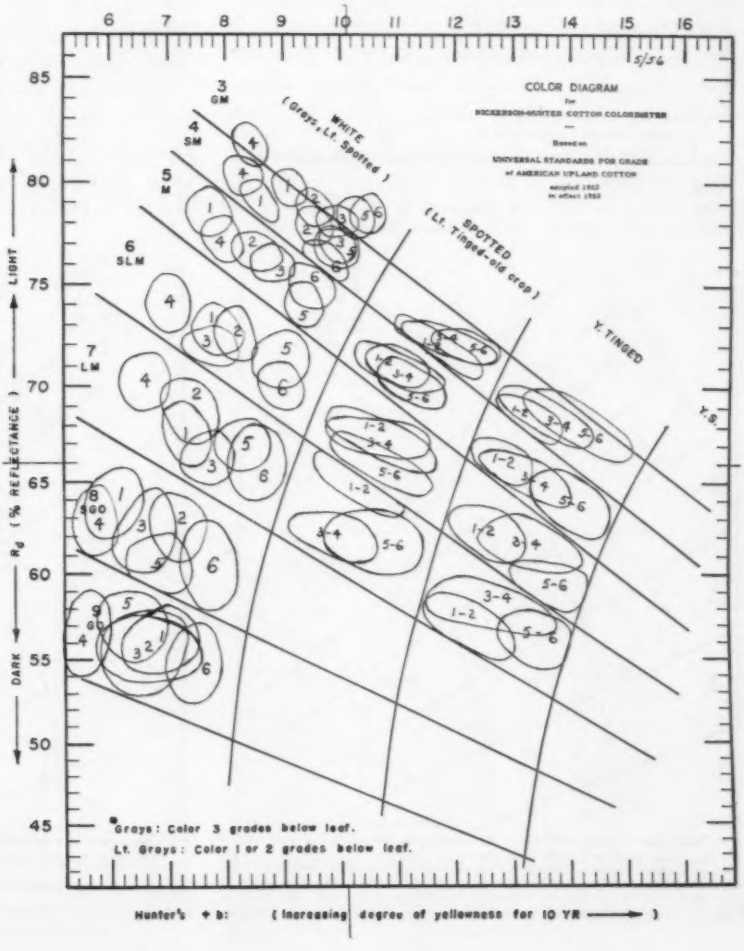


Fig. 1. Color diagram for the universal cotton grade standards. The color range of bales used in the 1956 standards and spotted guides is shown in the odd-shaped numbered enclosures. The number in each enclosure indicates the bale position in the standards box.

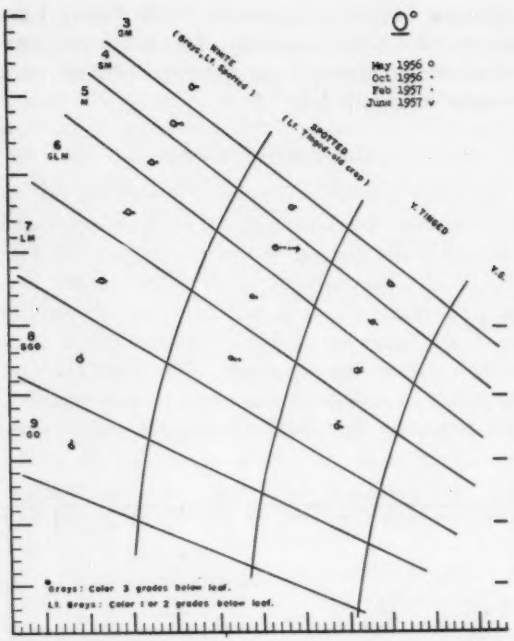


Fig. 2. Color change at 0° F. Storage for about 1 yr. (May 1956–June 1957) is indicated by the length of line from the center circle. No significant change is apparent.

age periods are shown by lines drawn from that circle. The center points are intended to be the same; therefore the length and direction of lines may be compared in order to learn the relative amount of change that occurred for each of the conditions reported.

Measurements were made in May 1956 when the standards were first put into storage. It was intended that a few sets be measured after 3 and 6 months to see how rapidly changes might occur, and all of them after 9 months and 1, 2, and 3 years. Measurements were made in August for the set held at 85° at Beltsville; in October for sets stored at Experiment; in February on all sets; in June on sets from Experiment; and in July on those from Beltsville. The date of each measurement is given on the diagrams, and the average results are plotted in sequence by date, so that it is possible to tell whether more change occurred in one period than in another.

The results are plotted on diagrams in order that the relation of the differences may be clear. For example, for the conditions in which considerable color change occurs during storage there is more change in high grades than in the low, and the rela-

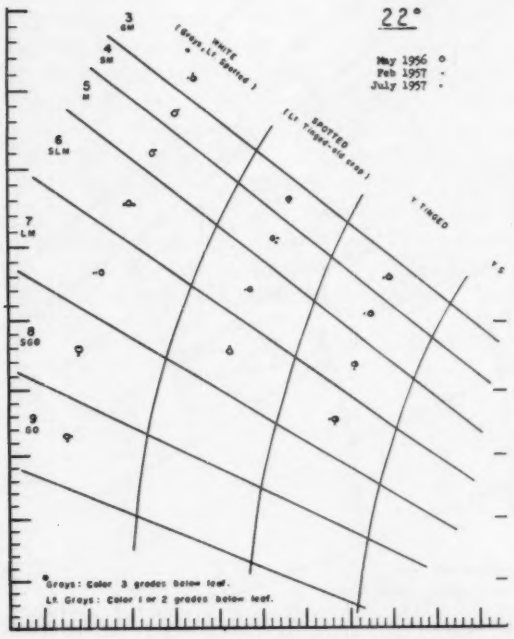


Fig. 3. Color change for storage at 22° F. is not significant in 1 yr.

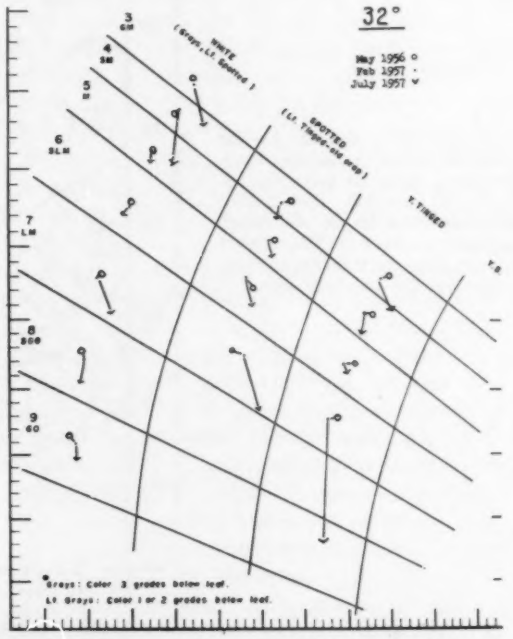


Fig. 4. Color change for storage at 32° F. is chiefly a darkening caused by microbiological action.

tive degree of change in Spotted and Tinged grades seems about the same as in the White grades.

Some of the color changes are not typical for cottons, but are caused by the molds and bacteria to which the samples were exposed as a result of storage with spoiling vegetables. Nevertheless, these extreme results are shown for contrast in order to demonstrate the color differences that can occur. For example, samples stored at 60° (Figure 8) changed so much in a 9-month period that they had to be discarded; even the boxes were in terrible condition, wet and moldy. In this 60° room sweet potato storage was being studied, and the organisms found in the cottons were those more natural to sweet potatoes than to cotton. However, even in this condition, which was so bad that several boxes were discarded without any attempt to measure them, the color change in the high grades was far more than in the low grades. (The dashed lines in Figure 8 represent duplicate sets of samples stored in another position in the same bin.) The direction of color change for this extreme condition is almost entirely in a vertical direction, indicating that the samples became very dark, instead of yellowing in the normal direction of color change during storage. Samples examined from sets at 50°, 70°, 85°, and 100°, all at high humidity, showed:[1] "*Penicillium* was present in considerable amounts on almost all of the samples, and significant but smaller amounts of an *actinomycete* and of *Fusarium moniliforme* were present. . . . The latter two organisms undoubtedly were on the fiber when it went into storage, while the *Penicillium* was almost certainly not." The *Penicillium* is a green or blue mold, and it was this which caused the darkening of color.

The general picture shown by Figures 2 through 9 makes it clear that little or no color change occurred in a year's time under 0° and 22°; from 32° through 38°, 40°, 45° the change seems a darkening caused by the presence of *Penicillium* on the fruits and vegetables stored in the uncontrolled high humidity of the storage bins; at 60° there may have been some yellowing, but this is overshadowed by the darkening caused by the *Penicillium;* at 85°, humidity high, even for the first 3 months of storage the color change

[1] From private communication March 18, 1956, from Dr. Paul B. Marsh, microbiologist, Cotton and Other Fiber Crops Section, ARS.

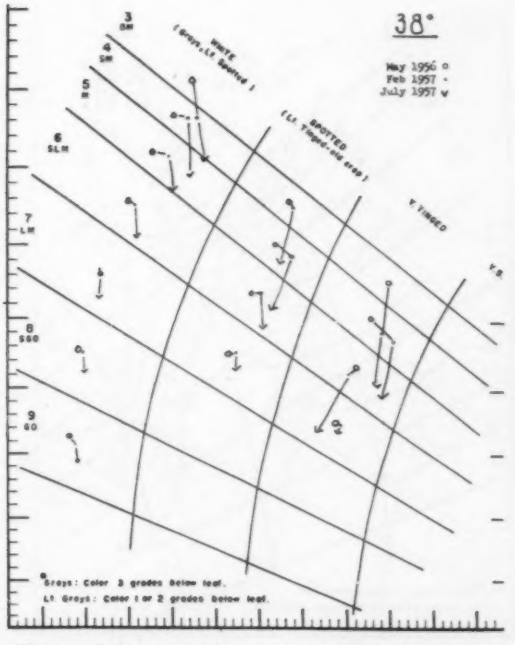


Fig. 5. Color change for storage at 38° F. is a slight yellowing, but chiefly a darkening caused by microbiological action.

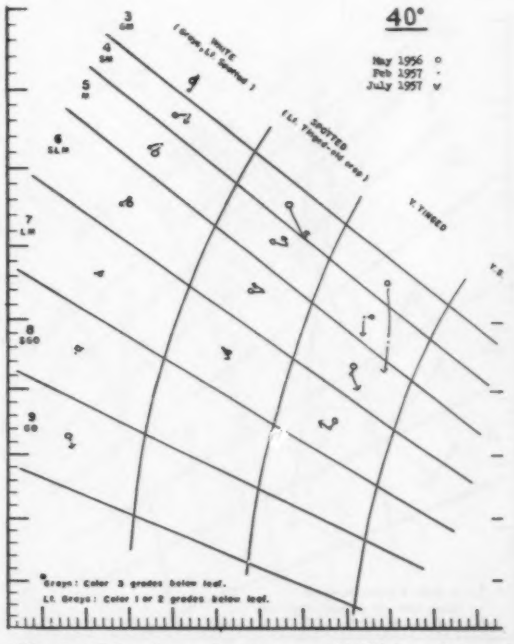


Fig. 6. Color change for storage at 40° F. is a slight yellowing, but chiefly a darkening caused by microbiological action.

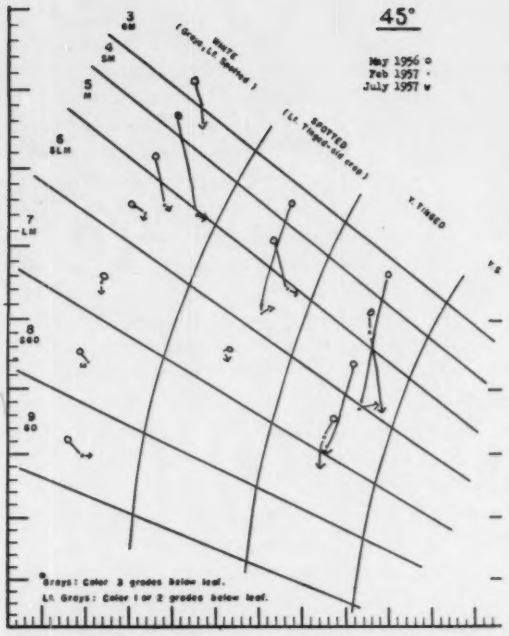


Fig. 7. Color change for storage at 45° F. is a slight yellowing, but chiefly a darkening caused by microbiological action.

is very considerable, the White grades yellowing into Spotted, the upper grades even into Tinged, with a darkening as well as yellowing for the Spotted and Tinged samples that were stored. Results shown in Figures 10, 11, and 12 are for storage at 50°, 70°, and 100° at two humidities, one held at 50%, the other uncontrolled, which measured about 85%–90%.

At 50°, Figure 10, little or no color change occurred in samples stored under low humidity, but change did occur in samples stored at high humidity. This change included some yellowing even within the first 9 months, but much of the change, particularly the darkening in the Spotted and Tinged grades, was undoubtedly caused by the *Penicillium*.

At 70° storage, Figure 11, there was a noticeable amount of yellowing at 50% humidity, especially in the higher grades, and a very great change at 85% humidity both in yellowing and in darkening of color. The change was considerable in the first 6 months of storage, and has continued since then.

At 100° storage, Figure 12, under 50% humidity, a noticeable yellowing occurred during the first 6 months that has continued since then. Under 90% humidity the color change was greater and occurred

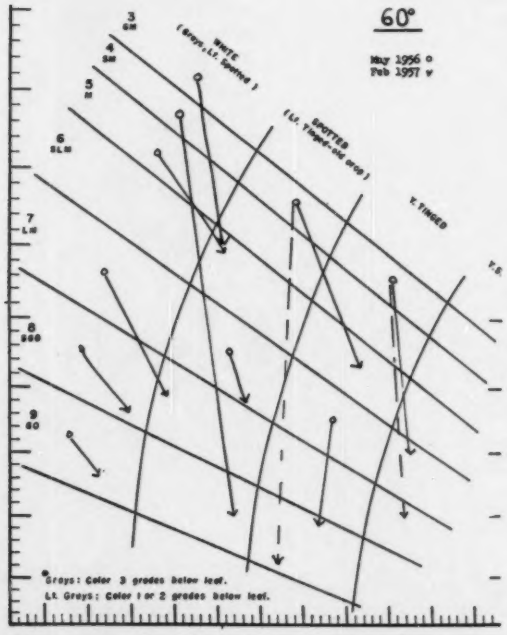


Fig. 8. Color change for storage at 60° F. is a severe darkening caused chiefly by high *Penicillium* contamination.

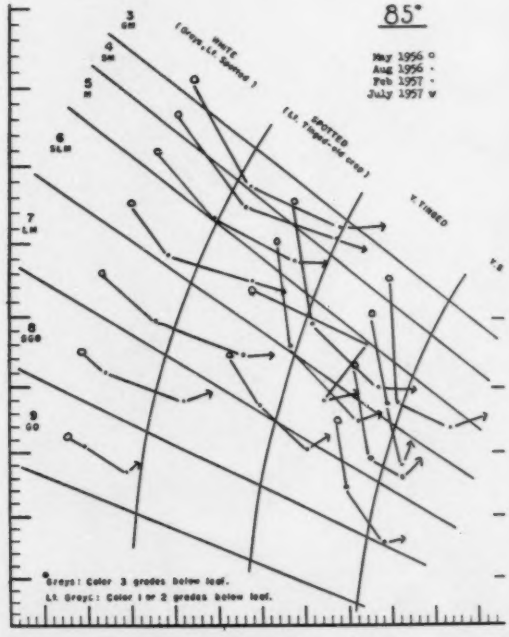


Fig. 9. Color change for storage at 85° F. is a considerable yellowing even after only 3 months of storage, with some darkening caused by microbiological action.

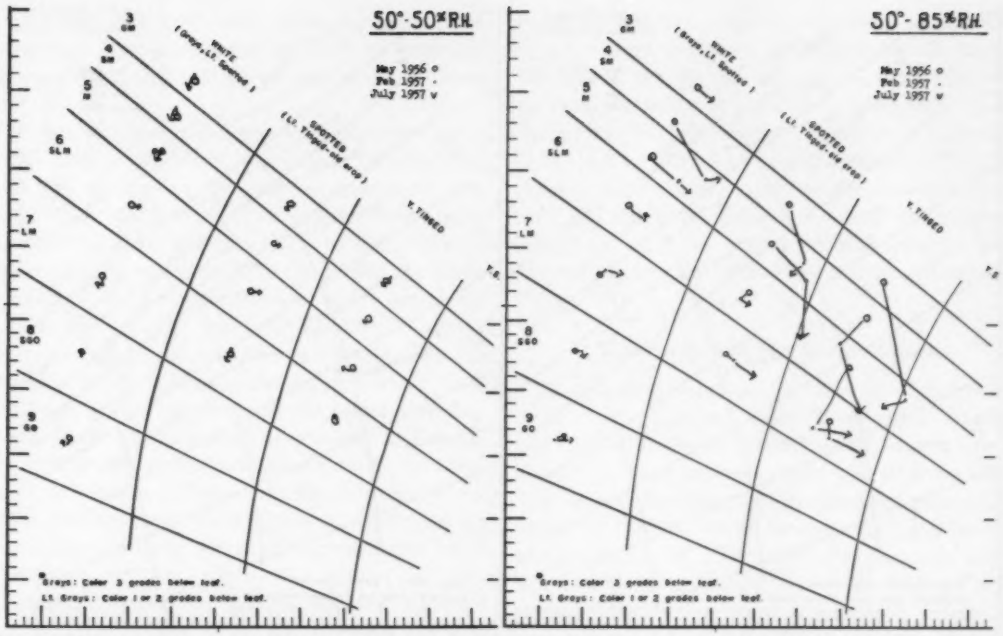


Fig. 10. Color change for storage at 50° F. At 50% relative humidity no significant change occurred in 1 yr., but at 85% relative humidity a significant yellowing occurred, greater for high grades than for low, with considerable darkening caused by microbiological action.

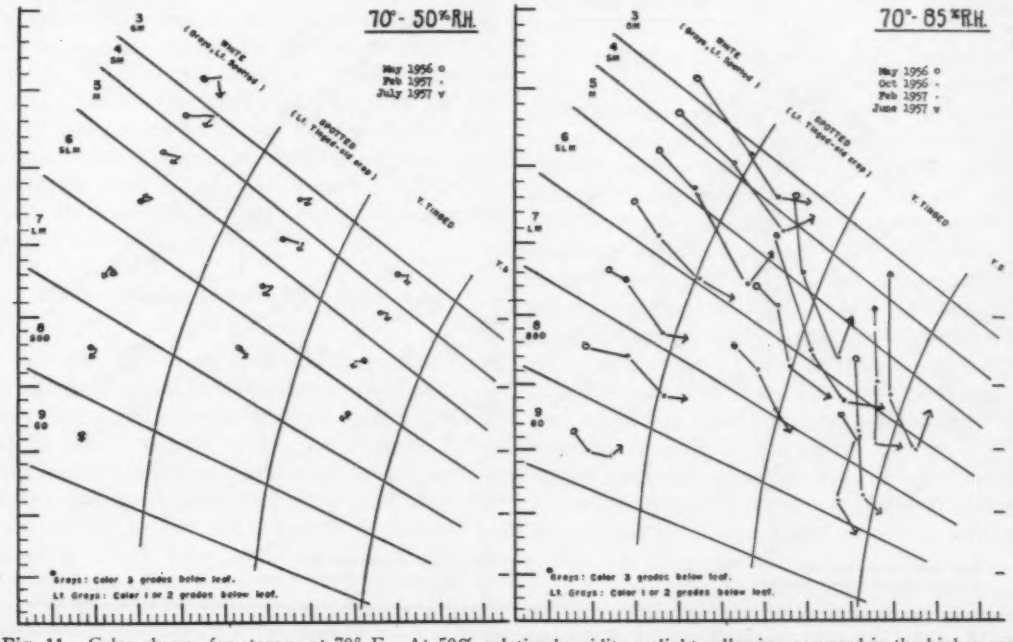


Fig. 11. Color change for storage at 70° F. At 50% relative humidity a slight yellowing occurred in the higher grades; a slight darkening was caused by microbiological action. At 85% relative humidity the color change was highly significant, with more yellowing in high grades than in low, and there was considerable darkening because of microbiological action.

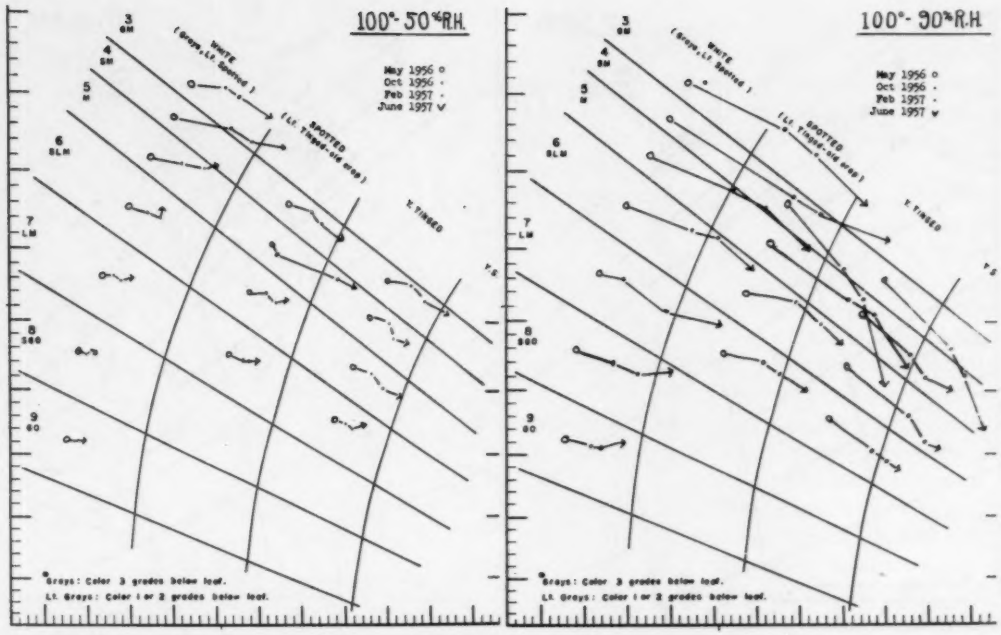


Fig. 12. Color change for storage at 100° F. At 50% relative humidity considerable yellowing occurred, more at high grades than at low; at 90% relative humidity there was an extraordinary amount of yellowing but little darkening.

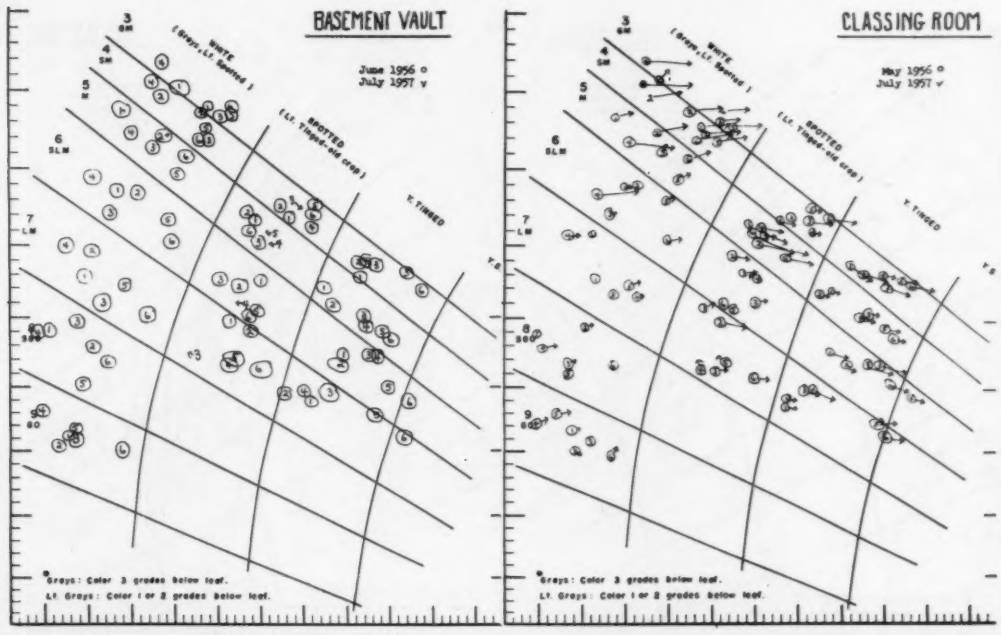


Fig. 13. Color change of individual samples of standards stored in basement vault and in classing room. In the first year no significant change occurred during storage in the vault, but significant though small changes occurred during storage in the classing room, particularly in the higher grades.

more rapidly. At 100° little darkening took place even at high humidity, since the 100° temperature quite evidently inhibited the growth of *Penicillium* mold.

The data in Figures 2 through 12 represent averages for each grade shown. In Figure 13 individual results are shown for two sets of standards, one stored in the basement vault in which extra standards are being stored between the 1956 and 1959 conferences and the other stored in the sixth floor classing room of the Cotton Division, where standards are made and passed. The bale position number for samples in each grade is circled when there was no measurable color change. Change in color is indicated by an arrow connected to the sample number. There are few and very short arrows on the diagram representing storage in the basement vault, but there are many changes indicated on the diagram representing samples stored in the classing room, and for the high grades these are significant changes for 1 year of storage.

Samples in the White grades represent different areas of growth, and since the question often is asked whether samples from one area change either more or more rapidly than samples from another area, a study was made of the differences in yellowness that developed under the several storage conditions for each sample by bale position number. The results of this study are shown in Figure 14. While it is not always true, and the differences are not always great, there does seem to be a tendency for the bale in No. 4 position to change more than those in other positions. Number 4 is the Western bale, usually the whitest bale in each box when the grades are first assembled.

Dollar Values Involved in Color and Grade Changes

The dollar values involved in changes that occur during storage are sometimes very considerable. To illustrate this in a manner in which price differences may be related directly to the size of grade differences involved in the various storage tests, Figure 15 shows prices for each grade posted on a color diagram similar to the one used in Figures 2–13. Prices for 500-lb. bales, based on 1956–57 average spot quotations for 14 markets for $1\frac{1}{32}$-in. cotton [2] [2], are entered for corresponding grades. For example, the average 1956–57 price for Good Middling $1\frac{1}{32}$-in. cotton is shown at \$176/bale. If bales of this grade change in storage to Good Middling Spotted, the value is reduced to \$153/bale; if they change to Good Middling Tinged, it is reduced to \$129, and to \$115 for Yellow Stained. The small changes involved in storage under favorable condition, and the large changes involved under adverse conditions, may be translated into relative dollar differences for the 1956–57 crop by reference to Figure 15.

[2] In 1956–57 11.7% of the crop was 1 in., 28% was $1\frac{1}{32}$ in., and 29.9% was $1\frac{1}{16}$ in.

Summary and Recommendations

In this study changes in color are reported for several sets of cotton from 1956 grade standards bales stored under 14 conditions of temperature and humidity. Temperatures varied from 0° F. to 100° F. The relative humidity was uncontrolled (and high at about 85%–90%) for all temperatures, and for three temperatures (50°, 70°, 100°) it also was kept at a relatively low humidity (about 50%). Color measurements are reported for samples before storage and periodically during storage up to a little over 1 year. The study will be continued for 3 years, but the picture regarding color change already has become so clear that results are reported for the first year.

Yellowing, or the deepening of the normal yellow color in cotton [8], is a matter of great concern in cotton storage. In spite of the darkening introduced into these particular test results by the unwanted and unusual introduction of *Penicillium* mold (through storage in chambers containing fruits and vegetables), from the results shown in the series of color diagrams, Figures 2 through 12, it seems clear that to hold changes in cotton color to a minimum in storage, refrigeration alone is not the answer. Storage under moderate temperatures (50°, perhaps even 60°) accompanied by controls that keep the relative humidity reasonably low (50% or less) should succeed in holding color change to a satisfactory minimum for at least 2 or 3 years. Where it is not feasible to control temperature, a control of humidity alone should succeed in reducing the degree of color change in storage that might otherwise take place within any given period.

It seems reasonable to suppose that there would be even less color change at humidities lower than 50%. However, if 8.5% is the accepted regain (the amount of moisture cotton will take up from a bone

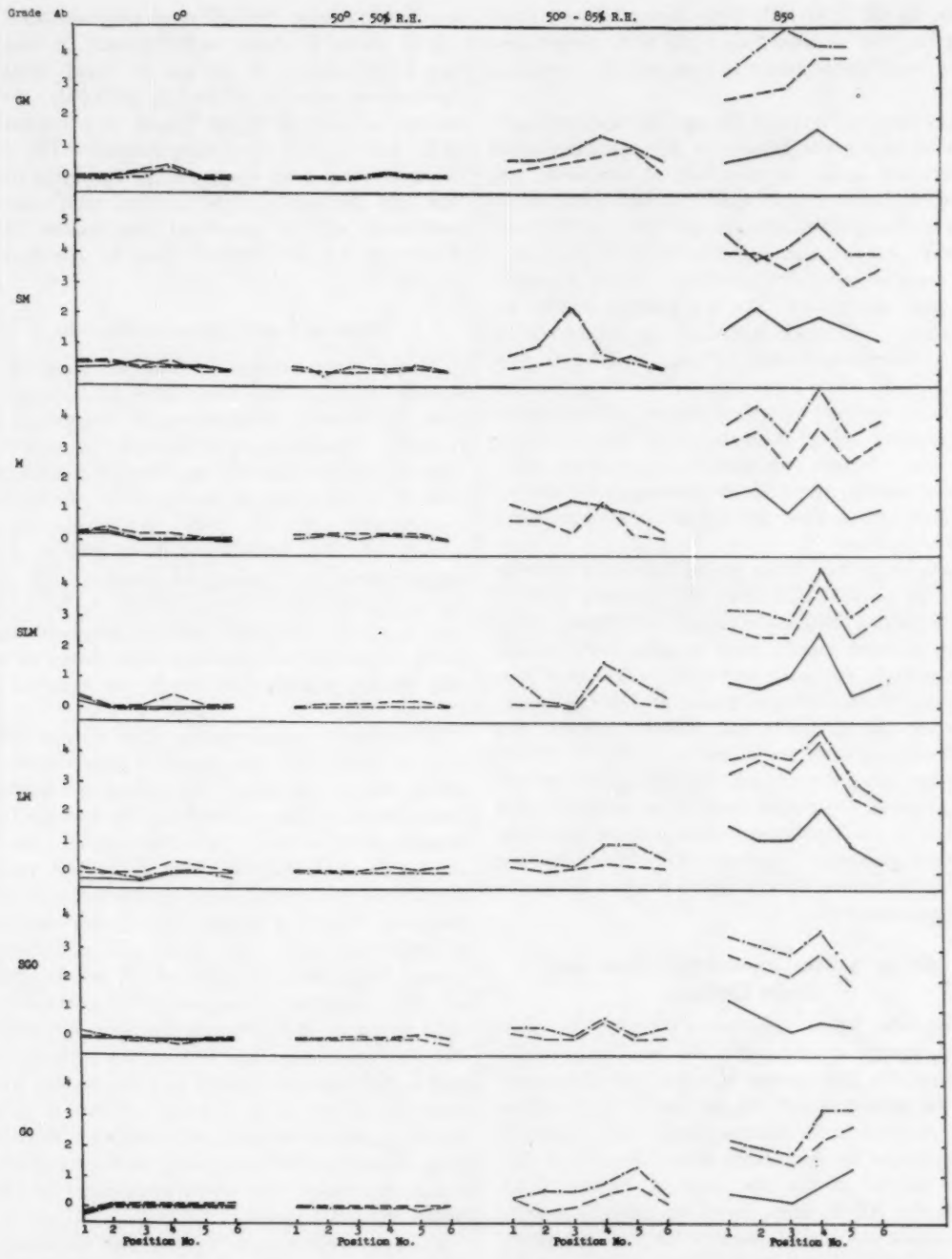
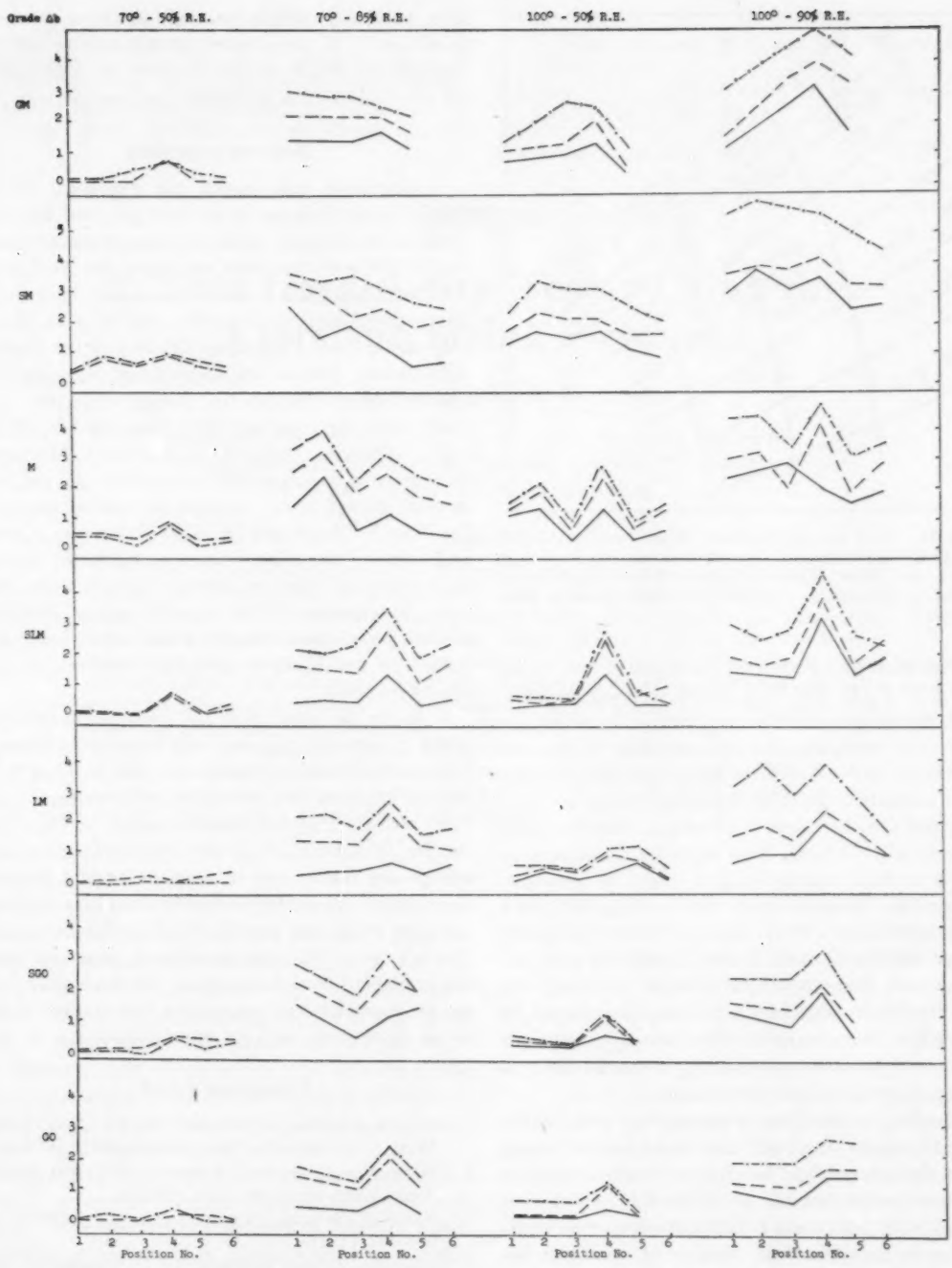


Fig. 14. Effect of storage on cottons from different areas. Cottons stored under several conditions of temperature and humidity; measurements in terms of differences in yellowness, Δb (measured on Cotton Colorimeter). Positions Nos. 1 and 2 are from the South Central area; No. 3, Southeast; No. 4, West; Nos. 5 and 6, Southwest. Code for storage:



—, 5 months; — —, 9 months; —·—, 13 months. A change of 2-3 units (Δb) is enough to cause a shift from one color class to another, as from White to Spotted; a change of 4 or 5 units is enough to shift the grade from White to Tinged.

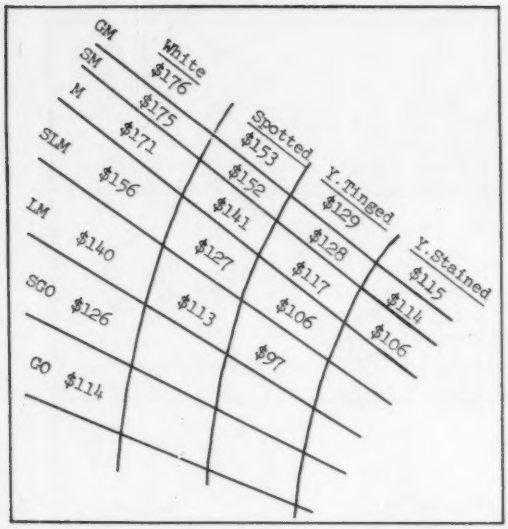


Fig. 15. Price per bale to nearest dollar, based on 1956–57 average spot quotations for 14 designated markets for 1 1/32-in. cottons, for White, Spotted, Yellow Tinged, and Yellow Stained grades shown (Good Middling through Good Ordinary).

dry condition) for cotton at the standard test conditions (70° F., 65% R.H.), then it seems undesirable to set storage specifications for relative humidity any lower than necessary, since the moisture in the cotton will be less at low humidities. Classing is done under conditions specified for many rooms at 73°/58%, and samples taken from cottons stored at 50% humidity will not be far from ready for normal classification in these rooms, while it might be necessary to condition samples taken from storage at much lower humidities. If it were *necessary* to specify storage conditions much lower in order to keep the cotton from changing, then it might be worth the extra trouble involved in conditioning samples and/or bales when they are taken from storage. But for the present, it seems satisfactory to specify 50% as the upper humidity limit for storage.

Therefore, on the basis of information provided by the test results reported, and until further work shows the advisability for further modifications, it is recommended that 50° F., 50% R.H. (with reasonably wide tolerances) be specified as the conditions to be maintained for storage of standards between conferences, and that dehumidification facilities be installed to keep the relative humidity at no more than 50% in space in which standards bales are stored after purchase. While these specifications are developed for U. S. Department of Agriculture use, the implications of this recommendation are important to all who hold cotton in storage for long periods.

Acknowledgments

Appreciation and thanks are expressed to Dr. H. C. Diehl, Director of the Refrigeration Research Foundation, through whom we located storage chambers of the sort that were necessary; to Dr. Harold T. Cook, Biological Sciences Research, Agricultural Marketing Service at Beltsville; and Dr. J. G. Woodroof of the Food Processing Division of the Georgia Experiment Station at Experiment, Georgia, for making space available in storage chambers used under their direction for other tests; to Mr. Q. W. Roop, Tobacco Division, Agricultural Marketing Service, who arranged for preliminary storage tests in cold storage space assigned to tobacco work; to Dr. Paul B. Marsh and Dr. Luis E. Gregory, Cotton and Other Fiber Crops Section, ARS, for making microbiological tests on selected samples from these tests; to members of Dr. Cook's staff at Beltsville and of the Cotton Division's staff in Atlanta who helped get our boxes of samples in and out of storage as required.

It might be noted that the storage chambers of AMS at Beltsville, together with those of the Georgia Experiment Station, provide the best facilities for a storage study of this sort that are anywhere available; we are therefore most grateful to Dr. Cook and Dr. Woodroof for the use of these chambers even though our results had to account for and discount the changes caused by storage in chambers contaminated by molds not usually found in cotton storage. We are sorry that uncontaminated chambers were not available for cotton storage, but since they were not we appreciate the cooperation that allowed us the use of those being used for other purposes.

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A Yarn Irregularity Tester Using the Vibrating String Principle

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Abstract

A type of yarn irregularity tester is described in which mass per unit length of a moving yarn is determined by a method in which a short section of the yarn is automatically maintained in resonant transverse vibration. The method makes possible a more direct determination of mass per unit length than is given by capacitance-type testers and leads to a calibration which is independent of the type of material being tested and the regain. The accuracy of the tester was examined by comparing its record with direct measurements of the mass per unit length of various yarns; the absolute agreement was very good, both for short-length (3 cm.) irregularities and mean count or linear density (over 1-m. lengths). In the former case worsted yarns from 22 to 88 tex (40's to 10's count) and having coefficients of variation (C.V.) ranging from 28% to 10% were measured with accuracies better than 0.3% of C.V. In the determination of mean count of similar yarns the accuracy averaged 1%.

Introduction

Numerous types of yarn and roving irregularity testers are commercially available [2, 3, 5, 25, 27], and the majority of these are based on the capacitance principle; i.e., the moving yarn or roving forms part of the dielectric of a capacitor. Variations in the mass per unit length of the yarn or roving give rise to variations in capacitance which may be measured in any of a number of ways and the result displayed on a calibrated chart or meter.

Such capacitance-type testers are in wide use and provide a satisfactory measurement for many purposes. They have, however, certain inherent disadvantages, among which is the possibility of substantial errors in irregularity measurement. These errors have been considered in recent papers by Hearle and Walker [11, 12], Mack [14], and Foster [9]. They arise from variability in dielectric constant in and between materials, and from other causes. Also, the accurate measurement of small fractional changes in capacitance is involved, which calls for a high degree of mechanical and electrical stability in the measuring circuits. This requirement is likely to be reflected in the cost of the instrument.

It appears therefore that there is scope for a tester whose calibration is independent of regain, of the structure and composition of the yarn, and of its location within the gauge. The paper describes a tester [4, 6] having these characteristics, with the additional advantages that it gives directly the mean linear density (count) as well as its variation and that no excessive mechanical or electrical stability is called for. The principle of operation is the same as that of the vibroscope [10, 16, 17, 20, 23]; i.e.,

measurement of the fundamental natural frequency f of transverse vibration of a section of yarn of length l held at a tension T. The mass m per unit length is then given by the relation [26]

$$m = \frac{T}{4l^2f^2} \quad (1)$$

Some important modifications and additions to normal vibroscope design have been made, however, to meet the requirements of a convenient irregularity tester.

1. The necessity for manual adjustment of the frequency of a driving oscillator with simultaneous visual observation of the amplitude of the yarn vibration to determine the resonant frequency is obviated by use of the method previously described by the authors [8, 15]; i.e., the section of yarn is made part of an electromechanical oscillator, so that the yarn is automatically maintained in vibration at its natural frequency in a somewhat similar fashion to a valve-maintained tuning fork.

2. Variations in frequency are converted electronically to voltages proportional to variations in linear density.

3. The yarn is fed through the measuring device at a constant speed, so that a continuous measurement of linear density along the yarn is maintained.

The latter feature has been employed in a vibroscope, described by Condon [7], for measurements on wire. The essential difference in the present design is the automatic maintenance of oscillations at the natural frequency. A U. S. patent recently granted to Seney [19] describes a yarn irregularity tester similar in principle to that discussed in this paper.

Description of Instrument

The form of apparatus found most satisfactory is illustrated in Figure 1. The yarn under test is transported at uniform speed past a measuring head by means of feed and take-up rollers. Speeds used in the experimental machine range from 10 to 200 cm./sec. The yarn is held at constant tension by a weight carried on a floating pulley, the value of the weight being set in accordance with the nominal linear density or count of the yarn being tested.

The measuring head comprises that section of the tester between two smooth pins, 5 cm. apart, over which the yarn is drawn. The section of yarn between the pins is maintained in resonant oscillation by a small transverse motion of one of the pins, which is attached to the armature of an electromechanical transducer in the form of an electromagnetic phonograph pickup. The motion of the oscillating yarn at its center is detected [13] by a photoelectric cell, and the output of the cell is amplified and fed back via a two-stage amplifier to the driving transducer in the proper phase and magnitude to sustain the oscillation. The frequency of oscillation is nominally 400 cycles/sec.

A valuable feature of the self-sustained vibroscope is that the measured quantity f, the resonant frequency of the yarn section, is to a good first approximation independent of the gain around the sustaining loop (provided the gain is sufficient to produce oscillation). This means that the amplifier gain and the sensitivities of the photoelectric cell and driving transducer are not critical in their stability requirements.

The amplitude of oscillation of the center of the

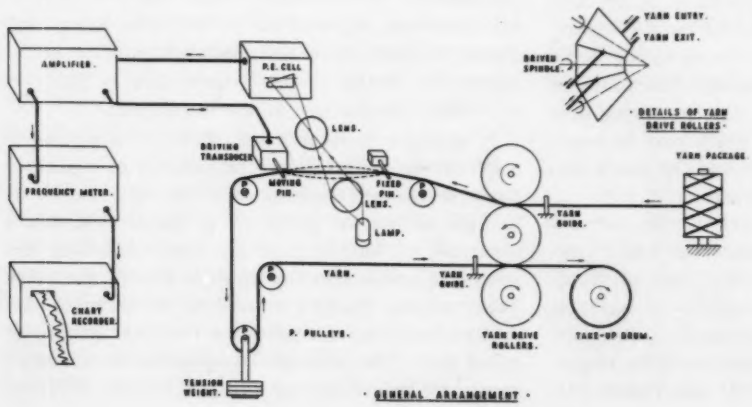

Fig. 1. General arrangement of C.S.I.R.O. yarn irregularity tester.

yarn is typically about 0.25 mm. The longitudinal motion of the yarn during test has a negligible effect on the frequency of oscillation with the yarn speeds normally used.

From some convenient point in the amplifier an electrical signal of frequency f, the resonant frequency of the yarn section, is taken to the indicator and recorder circuits. From Equation 1 it is seen that mass per unit length is proportional to $1/f^2$. If the tester is to provide a linear indication or record of mass per unit length, as is usually desirable, it is necessary to convert the quantity f into an electrical signal of magnitude proportional to $1/f^2$. This is done in a circuit using two "Miller integrator" stages [18] in tandem.

The output of the double-integrator circuit is fed to a recorder stage. The recorder is of the center-zero type. Variations in yarn mass per unit length from the nominal value cause linear departures of the record from the center line. Any one of three values of sensitivity can be selected, corresponding to full-scale deflections for variations in mass per unit length of ± 25, ± 50, and $\pm 100\%$.

The recorder in the present equipment has a chart width of 4 cm. Paper speeds are 0.1, 0.2, 0.5, 1.0, and 2.5 cm./sec.

The effect of a short irregularity on the frequency of vibration of the yarn section is greatest when the irregularity is at the center of the section, and decreases towards zero as the irregularity moves towards either pin. In other words, the central region of the yarn section is more important than the ends in determining the frequency. This means that the effective gauge length of the tester is less than the actual distance between pins. The effect can be analyzed fully [22] for any given form of irregularity along the section. However, as a working rule it can be taken that the effective gauge length is $\frac{3}{5}$ the actual length, or 3 cm. in the present design.

For any given nominal linear density or count of yarn there is an appropriate value of tensioning weight W to be used. This value may be calculated from a modification of Equation 1, inserting the nominal value of 400 cycles/sec. for f. For example, for a count of 40 tex (22's worsted) the weight is 13.2 g. In the laboratory tests a set of weights appropriate to the various counts being tested was used. When the proper value of W is used and the yarn is at its nominal count, the mean position of the record is at the center of the chart. A displacement of the mean position from the center shows that the yarn is off count by the indicated amount. The mean position of the record on the chart may be estimated visually or by an auxiliary meter associated with a smoothing circuit.

The value of tension used in the tester is small, producing a strain in the yarn of about 0.1%. Use of the same nominal frequency for all yarn counts means that all counts of yarn are subjected to the same (low) value of stress; this can be seen by rewriting Equation 1. We find that stress in yarn $= 4l^2 f^2 \rho$, where $\rho =$ density of the fiber substance, and therefore is constant for all counts and nearly constant for all yarns. The small strain produced in the length of yarn between feed and take-up rollers tends to accumulate there as the yarn runs through; to avoid this continuous lengthening, the take-up rollers are run at a slightly higher surface speed than the feed rollers.

In deriving Equation 1, certain simplifying assumptions are made; that the yarn is perfectly flexible and that the amplitude of vibration is infinitesimally small. These assumptions are not strictly valid. Also, because of friction at the pins, the tension in the yarn section under test will be slightly less than the tension $W/2$ due to the weight. For these reasons the mass per unit length will differ slightly from that given by Equation 1 [16, 20, 21]. However, these various sources of systematic error balance one another to some extent; some tests reported in the following section show that Equation 1 represents a very good approximation.

Tests

Reproducibility

A loop of yarn several meters long was run through the tester ten or more times. There was negligible difference between the records of successive passages of the yarn.

Comparison With Direct Weighing: Short-Length Variations

Records were obtained with the tester for a number of yarns of different types and counts. An 80-cm. length of each yarn was selected at random for comparison with the record. The 80-cm. length was carefully cut into 80 1-cm. lengths, each of which was weighed. Average values of the mass per unit length of the yarn over 3-cm. intervals were found

TABLE I

Yarn type	Correlation coefficient	C.V., % Cutting and weighing	C.V., % Tester
88 Tex (10's) worsted	.96	10.2	10.4
2/27.5 Tex (2/32's) worsted	.97	12.1	12.2
22 Tex (40's) worsted	.99	27.8	28.1

by averaging groups of three consecutive 1-cm. weights.

Figures 2a, 2b, and 2c show for various yarns the actual record (full line) on which are plotted (as circles) measured values of the mass per unit length. The agreement is clearly very good. It is emphasized that no correction factors have been applied to the record.

To provide a quantitative measure of the extent of agreement in the three cases, the correlation coefficient between indicated and true values has been calculated and is shown in Column 2 of Table I. Bearing in mind that the determinations of true weight are themselves subject to some random error arising from the cutting and weighing processes, one must regard the correlations as very high. To illustrate the effect of the tester errors on the coefficient of variation (C.V.) which would be estimated from the tester record (i.e., the indicated C.V.), Table I also shows in the case of each sample of yarn the actual C.V. and that determined from the record; the differences are seen to be negligible.

It will be noticed in the record of the heaviest yarn —88 tex (10's) worsted in Figure 2a—that there is a significant systematic deviation of the indicated from the true values. This arises presumably from the sources of systematic error discussed in the preceding section. In the case of this particular length of yarn, the average deviation amounts to about 4%, which is considerably greater than usually found, as will be seen from results described in the next section.

Longer-Length Variations

To find the accuracy of the tester for longer-length variations; i.e., for the determination of mean linear density or count, the measured weight of an 80-cm. length of several yarns was compared with the average weight shown by the record for the same length, the average being found by a planimeter method. The results are shown in Figure 3, which gives the results of 9 measurements on each yarn type. Again the agreement is seen to be very satisfactory. The mean deviation for all the measurements on each yarn type is as follows:

Yarn type	88 Tex (10's) worsted	2/27.5 Tex (2/32's) worsted	22 Tex (40's) worsted
Mean deviation, %	−1.1	−1.3	+0.7

Applications

The tester has two important advantages over capacitance-type testers: variations in linear density of yarn give rise to much larger percentage changes in the quantity being measured; and variations in linear density appear directly as variations of frequency, so that no special precautions are needed in regard to stability of circuit performance. For these reasons the tester circuitry is simpler and should be more reliable and cheaper to build.

The accuracy of the instrument for measurement of yarn irregularity is shown by the test results to be adequate for all practical purposes, while the additional indication of mean linear density or count is a useful feature. The use of the vibrating-string principle to determine linear density directly makes the calibration of the tester independent of the kind of material under test and of its regain. As a corollary, there is no calibration error in testing blended yarns.

The apparatus is suitable for use only with materials which approximate a flexible string in form. It is applicable therefore to practically all yarns and

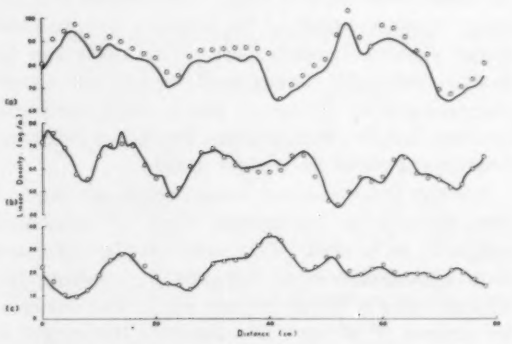

Fig. 2. Tester record of mass per unit length (continuous line) compared with measurements by cutting and weighing (circles). (a) 88 tex (10's) worsted yarn, 7 turns/in.; (b) 2/27.5 tex (2/32's) worsted yarn, 14 turns/in. (single), 11½ turns/in. (double); (c) 22 tex (40's) worsted yarn, 14 turns/in.

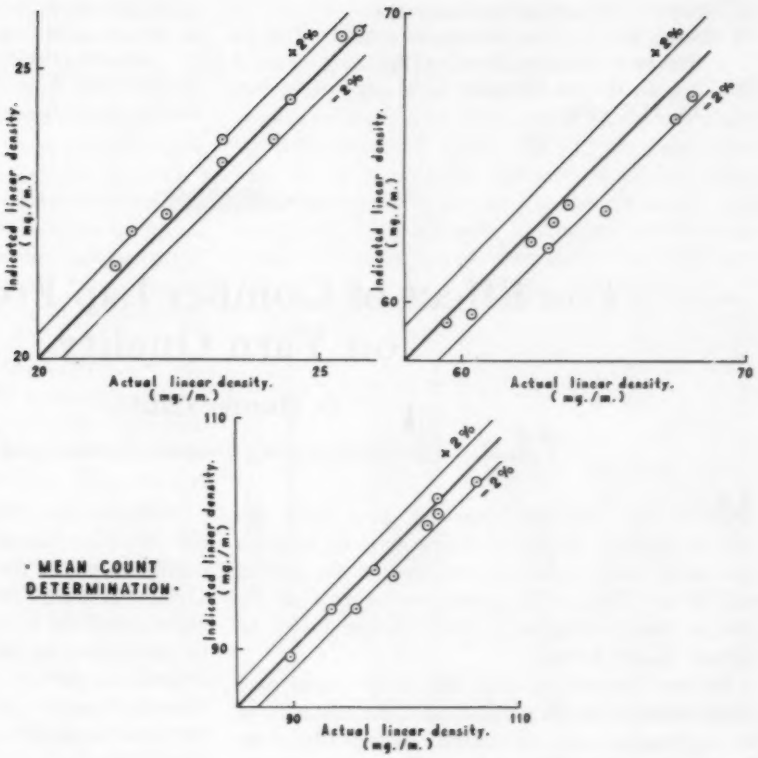

Fig. 3. Comparison of indicated and measured weights of 80-cm. yarn lengths, as check of accuracy of count indication.

monofilaments, but not to rovings, sliver, etc. Simple and cheap mechanical devices [1, 24] for measurement of the latter are available, and such a device easily could be combined with the present yarn irregularity tester where it is required to test the whole range of materials.

Acknowledgment

The authors wish to acknowledge the assistance of I. M. Stuart in discussions on the mathematics of the vibrating string; M. Beard of Radiophysics Laboratory, C.S.I.R.O., in the design of the double-integrator circuit; and A. B. Dehlsen in the construction and testing of the apparatus.

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The Effect of Comber Lap Preparation on Yarn Quality[1]

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MORTON and others showed some years ago that, in carding, the overwhelming majority of fibers have hooks on both ends, but the hook on the trailing end of the fibers in the sliver was larger than the one on the leading end. These larger hooks he termed "major hooks."

Further research showed that these hooks persisted through the drawing process; the efficiency of the combing process was materially affected by altering the direction of these fibers as they were presented to the comber. The experiments showed that if fibers were presented to the comber with the major hooks forward, a significant reduction in comber noil would result, with no adverse effect on yarn quality.

Morton thus recommended an even number of sliver preparation processes be used between the card and the comber so that the proper number of reversals in orientation of the major hooks in the fibers would take place.

Morton acknowledged, of course, that the savings depended largely upon the particular economics of the industry in which the experiments were tried; he further pointed out that his findings were restricted to long staple cottons. To quote his paper before the Combed Yarn Spinners Association in 1955, "There is no guarantee that with shorter and less expensive cottons, with American wage levels, and by comparison with existing American practice, worthwhile dollar savings will necessarily emerge."

[1] Presented at the Cotton Research Clinic of the National Cotton Council of America, Pinehurst, N. C., February 12, 1958.

Morton's conjecture was borne out soon after in our own experiments and in those conducted by him during his stay at the School of Textiles, N. C. State College, in 1955. While the details of these latter experiments are not yet available for release, it can be stated that the theory of using an even number of reversals did not work out on American cottons. American cottons and Egyptian cottons do not speak the same language, apparently.

In our experiments at Smyre, we tried three methods of lap preparation: one drawing followed by lap winding, two drawings followed by lap winding, and three drawings followed by lap winding.

Using the first method, the noil removed at the comber was 13.75%. Using the second method, noil dropped 2% to 11.75%, thus bearing out Morton's results. Yarn strength dropped off about 4%, however. There was no significant change in yarn uniformity as measured by evenness testers.

In the third trial, a series that oriented the sliver so that the fiber hooks were on the trailing end entering the comber (a circumstance that should have raised comber noil, according to Morton) the noil again dropped, this time to 10.0%. Strength dropped 7% below that for the one drawing process.

If the coarser American cotton fibers do not hook as readily as the Egyptian cottons, it can be assumed that the additional parallelization which results from increasing the number of drawing operations will present the fibers to the combing cycle in a fashion designed to reduce noil removal.

In our opinion, the yarn appearance was lower for

the three drawing operations. Aside from this, however, there are obvious economic disadvantages in increasing the number of drawing operations. A saving of 1% in noil reduces sliver cost 0.2¢/lb. This saving, if carried through to yarn, reduces yarn cost 0.37¢/lb. after the return from sale of noil is considered. To add an extra preparatory process would require an additional 40 deliveries of drawing, at about $1500/head. In addition, one additional worker per shift would be required, and amortization of these costs by the reduction in noil would require about five years, if the yarn quality made the installation worthwhile.

The progress of A. M. Smyre Manufacturing Company in improving comber lap preparation began with carding. The cards are kept in good mechanical condition, and the mill laboratory checks the nep count and evenness of each card the day after it is ground and returned to production. If the quality of the web and sliver does not meet standards, the card is rechecked until the trouble is corrected. Between grindings, all cards are rechecked periodically.

A 50-gr. sliver is produced from a 14-oz. lap. The company has adopted the speeds and settings for carding recommended by the School of Textiles, N. C. State College, modified in some cases to suit conditions, and through these settings and speeds has raised card production from 7 lb./hr. to 11 lb./hr. The flat speed was reduced from 3½ to 2½ in./min., with proportionate savings in waste. Some cards are equipped with metallic clothing; all will be converted to metallic in time. In addition to the savings in stripping and grinding time, there is 1¼–1½% less waste in carding and combing with no adverse effect on yarn quality.

The company uses Ideal drawing frames, 8 ends up, and produces a 43-gr. sliver with a draft of about 9.3. The front-roll speed is 300 ft./min., and it has been found desirable to use settings slightly more open than normal. Drawing at this relatively high speed requires maintaining equipment for processes prior to drawing in excellent mechanical condition,

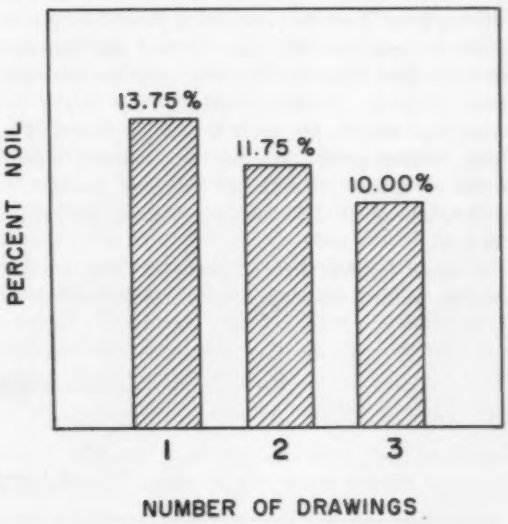

Fig. 1. Effect of number of drawing operations on percent noil.

TABLE I. Effect of Comber Lap Preparation

Yarn tests	Number of drawings 1	2	3
Percent noil	13.75	11.75	10.00
Skein break, lb.	126	124	122
Yarn no.	19.75	19.35	19.10
Break factor	2488	2399	2330
Twist multiplier	3.20	3.20	3.20
Uniformity:			
Brush % N. U.	65.0	64.7	65.5
Uster integrator	13.0	12.9	13.1

Comber Lap Preparation

Test no.		
1	1 Process drawing—lap winder	(2)
2	2 Process drawing—lap winder	(3)
3	3 Process drawing—lap winder	(4)

Note: All drawing processes are 8 ends up.

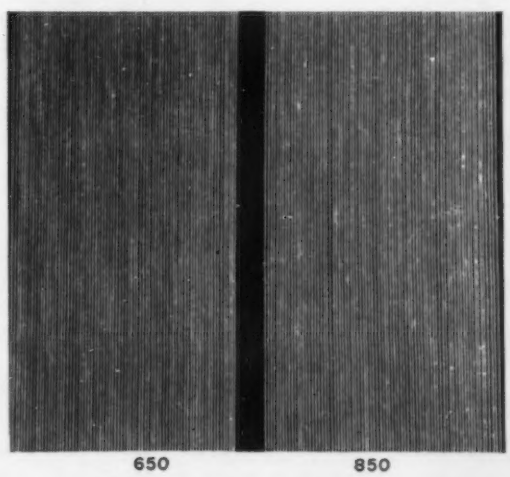

Fig. 2. Comber lap weight, gr./yd.

since mechanical troubles in these processes will show up in the quality of the drawing sliver. To locate mechanical troubles that show up in changes in the quality of the drawing sliver, good use has been made of the spectrograph instrument.

Drawing sliver is made into an 850-gr./yd. comber lap on a Saco-Lowell lap winder with 20 ends up. This lap is considerably heavier than the 600-gr./yd. lap made formerly using a sliver lapper/ribbon lapper preparatory system.

To obtain proper combing of this heavy lap, the half-lap is set closer to the cushion plate, the top comb is set to penetrate the mass deeper, and the most open needling thus far available from the manufacturer is used. Needles must be kept sharp and clean; flat needles are used in the top comb. Previous comber production, on the Nasmith, was 12 lb./hr. Present Saco-Lowell combers produce 53 lb./hr., and about 3% less long fiber is removed in the noil.

A lap of combed sliver is produced; this is passed through finisher drawing. Saco-Lowell 5-roll drawing, 16 ends up, with a front-roll speed of 186 ft./min. is used; a 60-gr. sliver is produced.

Roving frames are Saco-Lowell model FS-2, producing a 10 × 5 package. One recent change made in the roving process was to wrap the roving one extra turn around the presser; through the extra tension the weight of roving on the bobbin was increased by 40%, from 25 to 36½ oz.

Spinning is Whitin Casablanca in one mill and Gwaltney in the other; all roving is double creeled.

A vital part of the mill is the well-equipped laboratory, which develops improvements in manufacturing processes. There are four men in this laboratory (a ratio of 1/115 employees), who spend about half their time on quality control and the rest in research and development work which is aimed at improving quality, cutting costs, or increasing production.

Expenditures on research and development run close to 1.0% of sales, considerably higher than the average for the textile industry. This investment has always paid ample dividends.

Correction

In the article by Norman C. Davis in the April issue (Factors Influencing the Air Permeability of Felt and Felt-Like Structures), the equations at the bottom of page 320 should read:

$$v = \frac{-\Delta P}{\mu}\frac{1}{K} = \frac{\Delta P}{\mu}\frac{1}{K'X}$$

Therefore,

$$C = \frac{-\Delta P}{\mu K'}$$

The Use of Dial Gauges in Calculating the Results of Fibrograph Length Tests[1]

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Introduction

Cotton technologists in the U. S. Department of Agriculture have been interested in improved fiber length and length distribution measurements for a number of years. The slow and tedious array method has been used in our fiber laboratories since the late 1920's. When the Fibrograph instrument became available in the early 1940's, this method was substituted for the array in those tests in which speed of testing was essential. Since then, the two methods have been used alternately, with the array being used for detailed length distribution and the Fibrograph being used for speed of testing. During this time a number of other fiber length methods have been studied, such as the Uster staple diagram, short cut array, and truncated array. None of these methods, however, has provided the desired combination of reproducible length and length distribution results and speed of testing. The work on which this progress report is based represents another attempt to obtain such a combination of speed and accuracy.

Description of Method

For the method described, two dial gauges mounted on the Fibrograph are used to obtain length results instead of using the Fibrogram drawn by the instrument. One of these gauges is mounted to measure the movement of the comb carrier and the other is mounted to measure the movement of the card carrier. These measurements represent the relative length of fiber and the relative number of fibers on the Fibrogram, respectively. Measurements with these gauges at a number of places on the Fibrogram are used to calculate the Fibrograph results instead of using the normal graphic method (Figure 1).

The test specimens are prepared and the combs are placed in the instrument in the usual manner. The instrument is then balanced and the number of fibers dial reading (a) is recorded for 0 length of fiber dial reading. The comb carrier is then raised to 0.25 length of fiber dial reading, the instrument is balanced, and the number of fibers dial reading (b) is recorded. The mean length of fiber dial reading is then calculated from the (a) and (b) readings by applying the following formula:

$$\text{Mean length dial reading} = 0.25a/(a - b)$$

Both a table and a nomograph have been prepared so that this calculation can be made without delaying

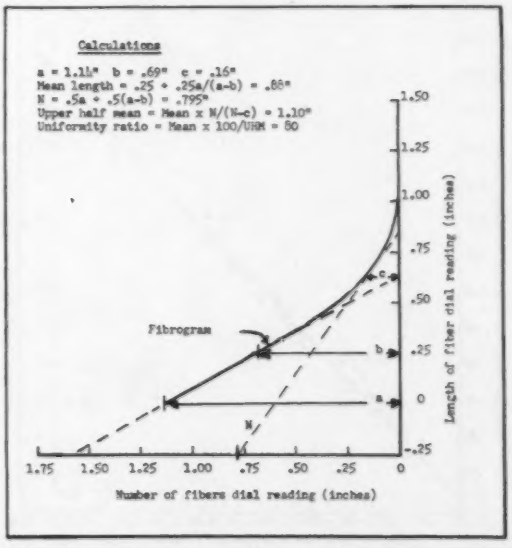

Fig. 1. Dial Fibrograph method of calculating the standard length and length uniformity results.

[1] Presented at the Cotton Research Clinic of the National Cotton Council of America, Pinehurst, N. C., February 13, 1958. The experimental work on which this presentation is based was delegated to the Cotton Division, AMS, by the Marketing Research Division, AMS, and supervised by Samuel T. Burley, Jr., formerly of the Cotton Division but now of the Marketing Research Division. Helpful assistance was received from Roger L. Yoakum of the Cotton Division and from George E. Gaus and Frances Carpenter, now of the Marketing Research Division.

the operation of the instrument. The comb carrier is then raised to the mean length of fiber dial reading, the instrument is balanced, and the number of fibers dial reading (c) is recorded. The specimens are then removed from the instrument in the usual manner.

Similar readings are taken for four specimens and are averaged as a basis for the calculation of the Fibrograph results for the samples. These calculations are made by applying the following formulas:

$$\text{Mean length} = 0.25 + 0.25a/(a - b)$$
$$= 0.25 + \text{mean length dial reading} \quad (1)$$

$$N(\tfrac{1}{2}\text{ no. of fibers}) = 0.5a + 0.5(a - b) \quad (2)$$

$$\text{Upper half mean} = \text{mean length} \times N/(N - c) \quad (3)$$

$$\text{Uniformity ratio} = \text{mean length} \times 100/\text{upper half mean length} \quad (4)$$

Some preliminary work was performed in an attempt to obtain more detailed distribution results in addition to the method described above. In this work, number of fibers dial readings were taken at regular intervals on the length of fibers axis. Percentage of fibers in the various length groups was calculated from these readings. Neither the reproducibility of these results nor their relationship with the results obtained by the array method, however, was very good. The details of the methods used therefore are omitted in this presentation.

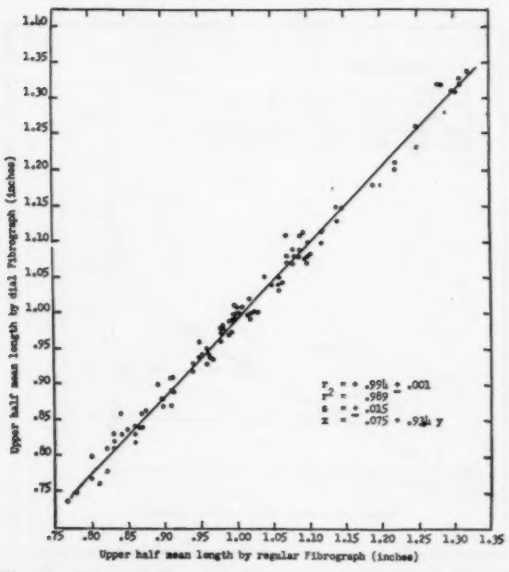

Fig. 2. Upper half mean results by both the dial and the regular Fibrograph methods for 100 samples of cotton.

Results of Comparative Tests

Results of preliminary tests using this method indicate that the reproducibility of the upper half mean length is practically the same for both the dial and the regular Fibrograph methods. The variability of the mean length, however, is approximately one-half as large for the dial method as compared to that for the regular method. This provides more accurate uniformity ratio results for the dial method. Also, the time required to perform the tests is approximately the same for the two Fibrograph methods.

In addition to these preliminary tests, two series of cottons for which other test results were available were tested routinely. One of these series consisted of 100 samples for which Fibrograph results were available from four laboratories. The other series consisted of 60 samples for which array, Fibrograph, and spinning test results were available. The data obtained from these two series of tests provide a good basis for evaluation of the dial method.

The length results for the 100 samples indicate that the relationship between upper half mean lengths by the two Fibrograph methods is very good (Figure 2). This relationship may be described by the following simple correlation results:

Coefficient of correlation $(r) = + 0.994 \pm 0.001$
Coefficient of determination $(r^2) = 0.989$
Standard error of the estimate $(s) = \pm 0.015$
Regression equation for $x = 0.075 + 0.934\ y$

where x = U.H.M. by regular Fibrograph and y = U.H.M. by dial Fibrograph.

The length distribution results for the 100 samples indicate that the relationship between uniformity ratios by the two Fibrograph methods is rather poor (Figure 3). This relationship may be described as follows:

Coefficient of correlation $(r) = + 0.668 \pm 0.056$
Coefficient of determination $(r^2) = 0.447$
Standard error of the estimate $(s) = \pm 1.00$
Regression equation for $x = 39.372 + 0.510\ y$

where x = U.R. by regular Fibrograph and y = U.R. by dial Fibrograph.

At the time this study was being performed, the truncated or cut-off array was also being studied. This method is the same as the regular array, except that the fibers shorter than $\frac{1}{2}$ in. are not sorted into different length groups. Also, mean length of fibers $\frac{1}{2}$ in. and longer, coefficient of variation of fibers

½ in. and longer, and percentage of fibers ½ in. and longer are substituted for the regular array results. The correlation of the truncated array results with spinning test results was slightly higher than it was for the regular array. We therefore compared the dial Fibrograph results with the truncated array results in our evaluation.

The length results for the 60 samples indicate that the relationship between upper half mean length by the dial Fibrograph and mean length by the truncated array is good (Figure 4). This relationship may be described by the following simple correlation coefficients:

Coefficient of correlation $(r) = + 0.906 \pm 0.023$
Coefficient of determination $(r^2) = 0.821$
Standard error of the estimate $(s) = \pm 0.029$
Regression equation for $x = 0.163 + 0.816\ y$

where x = mean length by truncated array and y = upper half mean by Fibrograph.

The length distribution results for the 60 samples indicate that the relationship between uniformity ratio by the dial Fibrograph and the coefficient of variation of fibers longer than ½ in. by the truncated array is also good (Figure 5). This relationship may be described by the following simple correlation results:

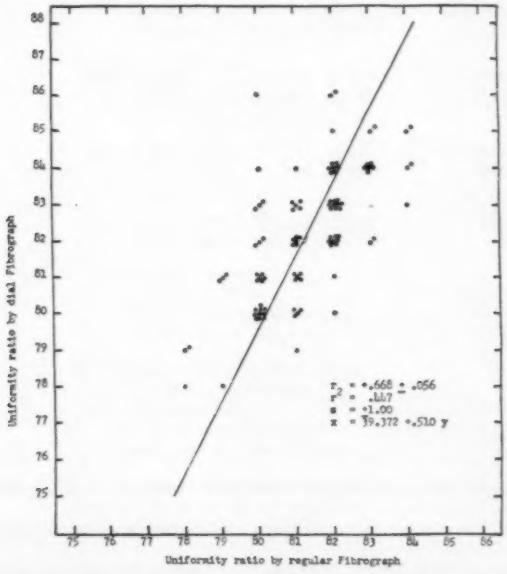

Fig. 3. Uniformity ratio results by both the dial and the regular Fibrograph methods for 100 samples of cotton.

Coefficient of correlation $(r) = 0.874 \pm 0.031$
Coefficient of determination $(r^2) = 0.763$

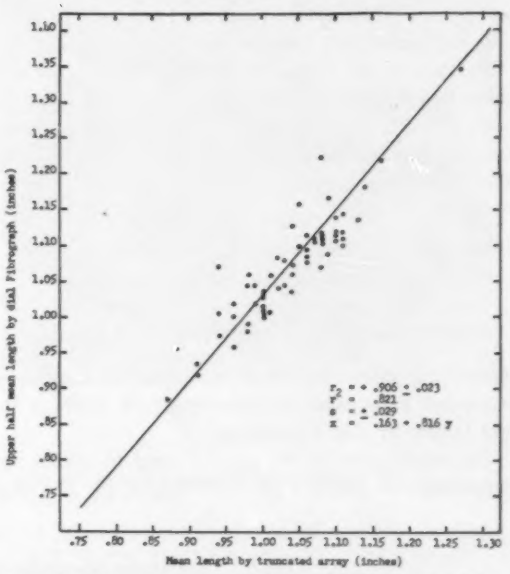

Fig. 4. Upper half mean length results by the dial Fibrograph and mean length results by truncated array for 60 samples of cotton.

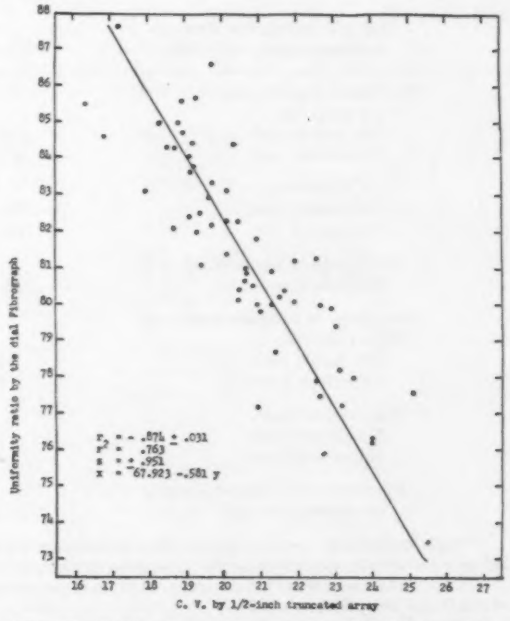

Fig. 5. Uniformity ratio results by dial Fibrograph and coefficient of variation results by ½ in. truncated array for 60 samples of cotton.

Standard error of the estimate (s) = ± 0.951
Regression equation for $x = 67.923 - 0.581\ y$

where x = coefficient of variation by truncated array and y = uniformity ratio by dial Fibrograph.

Percentage of fibers shorter than ½ in. also was calculated for the dial Fibrograph, but its relationship with percentage of fibers shorter than ½ in. for the truncated array was rather poor. This relationship may be described by the following simple correlation results:

Coefficient of correlation (r) = + 0.502 ± 0.097
Coefficient of determination (r^2) = 0.252
Standard error of the estimate (s) = ± 2.62
Regression equation for $x = 64.465 + 0.274\ y$

where x = percentage of fibers ½ in. and longer by truncated array and y = percentage of fibers ½ in. and longer by dial Fibrograph.

An explanation of the method used to obtain the percentage of fibers ½ in. and longer for the dial Fibrograph was omitted because of the poor relationship with the array method and because percentage of fibers ½ in. and longer was insignificant in the correlation with spinning test results. The length and spinning test results for the 60 samples indicate that the correlation between length and spinning test data is approximately the same for the dial Fibrograph and truncated array methods, but is much lower for the regular Fibrograph method (Table I). The dial Fibrograph method of length and length distribution explains approximately 68% of the variance in yarn strength as compared to 64% explained by the truncated array and 47% by the regular Fibrograph. For yarn appearance, it explains 12% of the variance for 22s and 40% for 50s as compared to 14% of the variance explained for 22s and 40% for 50s by the truncated array. Less than 2% of the variance in yarn appearance of 22s yarn is explained by the regular Fibrograph method. The dial Fibrograph method explains 29% of the variance in picker

TABLE I. Correlation Results for Three Methods of Length Measurements with Spinning Test Results for 60 Samples of Cotton

Multiple correlation statistic and dependent variable*	Comparative multiple correlation results for specified fiber length test methods: Dial Fibrograph†	Truncated array‡	Manual Fibrograph**
Correlation coefficient:			
Yarn strength:			
22s carded yarn	.833 ± .040	.807 ± .045	.684 ± .069
50s carded yarn	.821 ± .042	.794 ± .048	— —
Yarn appearance:			
22s carded yarn	.351 ± .114	.371 ± .112	.113 ± .128
50s carded yarn	.632 ± .078	.633 ± .078	— —
Percentage of waste removed at the picker and card	.535 ± .093	.555 ± .090	— —
Percentage of variance explained:			
Yarn strength:			
22s carded yarn	69.4	65.1	46.8
50s carded yarn	67.4	63.0	—
Yarn appearance:			
22s carded yarn	12.3	13.7	1.3
50s carded yarn	39.9	40.1	—
Percentage of waste removed at the picker and card	28.6	30.8	—

* Skein strength of yarn in pounds, yarn appearance grades as an index, and amount of waste removed at the picker and card as a percentage were used as the dependent variables.

† Upper half mean length, uniformity ratio, and percentage of fibers longer than ½ in. were used as independent variables for the Dial Fibrograph method.

‡ Mean length, coefficient of length variability, and percentage of fibers longer than ½ in. were used as independent variables for the truncated array method.

** Upper half mean length and uniformity ratio were used as independent variables for the manual Fibrograph method.

and card waste as compared to 31% explained by the truncated array method. The inclusion of percentage of fibers ½ in. and longer by the dial Fibrograph as a third item in the analyses may slightly exaggerate the differences between the two Fibrograph methods in the comparison above. Beta coefficients, however, indicate that our measures of percentage of fibers ½ in. and longer are statistically insignificant in all the relationships studied (Table II).

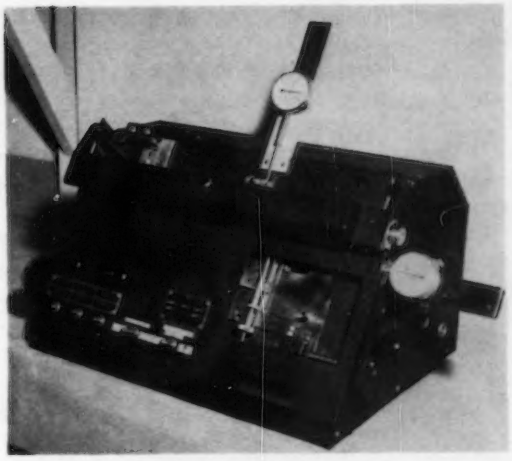

Fig. 6. The dial Fibrograph.

Summary

The advantages of the dial Fibrograph method are summarized as follows:

1. The variability of the mean length is only one-half as large as it is for the regular Fibrograph

TABLE II. Correlation Beta Coefficient Results of Three Methods of Length Measurements with Spinning Test Results for 60 Samples of Cotton

Dependent and independent variables	Comparative multiple correlation Beta coefficients for specified fiber length test methods		
	Dial Fibrograph	Truncated array	Manual Fibrograph
Yarn strength:			
22s carded yarn:			
Length of fibers†	+.812 ± .080	+.781 ± .108	+.686 ± .100
Uniformity of fiber length‡	+.587 ± .157	−.419 ± .122	+.335 ± .100
Percentage of short fibers**	*−.103 ± .153	*−.081 ± .140	— —
50s carded yarn:			
Length of fibers	+.808 ± .082	+.864 ± .109	— —
Uniformity of fiber length	*+.430 ± .162	*−.380 ± .127	— —
Percentage of short fibers	*+.002 ± .157	*−.205 ± .144	— —
Yarn appearance:			
22s carded yarn:			
Length of fibers	*+.089 ± .135	*−.088 ± .169	*−.060 ± .136
Uniformity of fiber length	*+.241 ± .265	*−.190 ± .192	*+.082 ± .136
Percentage of short fibers	*+.126 ± .258	*+.234 ± .219	— —
50s carded yarn:			
Length of fibers	*−.050 ± .111	*−.211 ± .139	— —
Uniformity of fiber length	+.783 ± .220	*−.449 ± .161	— —
Percentage of short fibers	*−.205 ± .214	*+.232 ± .183	— —
Percentage of waste removed at the picker and card:			
Length of fibers	−.475 ± .120	−.376 ± .150	— —
Uniformity of fiber length	*+.174 ± .239	*−.274 ± .174	— —
Percentage of short fibers	*−.229 ± .233	*−.286 ± .197	— —

* These values are statistically insignificant, being less than 3 times their standard error.

† Upper half mean length for both Dial and manual Fibrograph methods and mean length for the truncated array method were used as the independent variables.

‡ Uniformity ratio for both Dial and manual Fibrograph methods and coefficient of length variability for the truncated array method were used as the independent variables.

** Percentage of fibers ½ in. and longer for both the Dial Fibrograph and truncated array methods were used as the independent variables.

method, which provides more accurate uniformity ratio results.

2. The general level of length and length uniformity results is approximately the same as for the regular Fibrograph.

3. There is a good correlation between both the length and the length distribution results and similar results for the array method.

4. The correlation of the results with spinning test data is much higher than it is for the regular Fibrograph method and is practically the same as it is for the array method.

Our study shows that the dial Fibrograph provides a good method of measuring fiber length and length uniformity, but we do not consider it to be the ultimate or final method. We can visualize such further improvements as automatic operation by merely pushing buttons, automatic computers, mechanical preparation of the test specimen, and other developments. Until such improvements materialize, however, we believe that this dial method is a practical one which provides a better combination of speed of testing and accuracy of results than do other available methods.

An Optical Measurement of the Trash Content of Ginned Cotton[1]

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Abstract

Current methods of evaluating the amount and particle size of trash in ginned cotton are reviewed and the need for simplicity and speed in obtaining a measurement of trash content is evidenced.

The authors discuss a new optical–electronic method of scanning a classer's sample to record both particle size and number of particles. The sum of the areas in the sample which are darker than the background is taken as a percent of the total area scanned.

The method utilizes a closed circuit television camera, which in a fraction of a second traverses the face of a sample and reads out the desired information directly. Results of tests designed to prove the reliability and validity of the method are shown and a comparison to current methods of evaluation is given.

Diagrams of the optical and computing systems used in the method are explained in detail.

Introduction

Many improvements in cotton merchandising and mill processing are based on measurements of specific properties of the raw fiber. The papers of the Cotton Research Clinic [1] on the subjects of nep potential, length characteristics, maturity, and Micronaire readings amply illustrate this point. These values, which are being applied at all levels of cotton marketing and manufacturing, enable the intelligent selection of cottons for maximum utilization of special properties. In general, the instrumental values placed on fiber properties supplement the information supplied by the cotton classer.

Linton [4] defines the cotton classer as "the expert who actually classifies raw cotton for color, grade, quality, staple length, tinges, and other characteristics." Linton continues, "Assets the classer should possess are keenness of eyesight, fine sense of touch, and years of actual experience." Obviously, the

[1] Presented at the Cotton Research Clinic of the National Cotton Council of America, Pinehurst, N. C., February 14, 1958.

multiple values involved in classing cotton by hand cannot easily be duplicated in a single instrument. Hence we currently have a number of instruments designed to measure specific parts of the total evaluation.

When the elements of classing are broken down there appear to be several phases which can best be measured optically. Nickerson [5] describes apparatus which will measure color and reflectance in this fashion.

Trash in cotton has required mechanical separation by the Shirley [6] analysis system of measurement, although Kirkwood [3] suggests the use of the difference in dielectric constants of cotton and trash as a possible means of evaluation. Neither method is capable of describing the particle size of leaf and trash, which is critical to a total weighing of value. Pepper trash obviously creates removal problems which are not present with large leaf and twig contaminants.

Another visual aspect of cotton examination is the appearance of bright spots known as preparation. These lighter than background spots spell trouble to the classer and are considered in his call.

In the foregoing description of some aspects of classing cotton it becomes obvious that the classer takes into consideration many elusive and interrelated properties. An apparatus designed to measure yellowness of color, reflectance, preparation, trash volume, and number of trash particles is described below. Proving the validity and reliability of the instrumental measurements is the subject of this paper.

Apparatus

The device referred to hereinafter as the Cotton Classer will automatically read and record the following parameters:

1. Preparation—a percentage figure based on lighter than background areas.
2. Leaf or trash—a percentage figure based on darker than background areas.
3. Particle size—a relative numerical designation of the particles in the measured area.
4. Reflectance—a percentage value in relation to total reflectance.
5. Color—in terms of yellowness or off-whiteness in percent.

The sample of cotton to be tested is placed over the $3\frac{1}{2} \times 4\frac{1}{2}$-in. viewing window of the instrument. The lower face of the sample is illuminated by means of four prefocussed and voltage regulated incandescent bulbs, the angle of incident light being 20° with the sensing elements at 90°. The sensing elements consist of four vacuum type photocells, appropriately filtered and balanced in the tristimulus coordinate. The fifth sensing element for the preparation and leaf counting is a closed circuit TV camera. The light reaching the vidicon camera tube is filtered by means of a blue filter which passes light in the blue region. The vidicon transducer is most efficient in this range, and extraneous light frequencies which may cause interference in the output of the vidicon are eliminated.

A manual starter button is depressed by the operator after the cotton sample is in place over the viewing window. The indexing switch moves to the preparation count position. The signal from the TV system consists of positive going pulses for whiter than background information and negative going pulses for darker than background pulses (leaf).

This composite information is fed to a keyed clamp which establishes the background, or base line level. This information is then fed to a separator which splits this composite information into two channels, positive going "prep" and negative going leaf. The negative pulse is inverted to positive going leaf. The selector switch channels the prep information to the second of two gates. The first gate is controlled by the blanking information from the camera system. The first gate allows 1-Mc. clock pulse bunches to appear at the second gate only during trace time of the camera system. Output from the second gate occurs only during the time that a prep pulse is available. The duration of open gate operation depends upon the size of the prep, or whiter than background spot. The gated clock pulses are then fed to an electronic counter which totalizes and reads out the result in percentage, after precisely four TV scannings of the image. The start and stop control is provided by binary dividers which are triggered by the 60^n vertical scanning frequency of the TV system. The output from binary is 7.5^n.

Since a single time base is required to supply television synchronization signals and the clock frequency, a master clock is provided. This is to insure that the length of time of one frame does not vary from instant to instant due to line frequency variations. The clock is fundamentally a crystal oscillator, whose frequency is multiplied and divided.

The television period is selected from the submultiple of 31.5 Kc. This frequency is divided by a series of phantastrons in the camera synchronization generator until the 60th vertical frequency is reached. Thus the complete sequence of operation is locked to the master clock.

After 0.1 sec. has elapsed, the prep reading has been totalized and is fed to the printer, which records the numerical percentage results on paper tape; the machine automatically indexes to the leaf reading. In this position the positive going leaf information from the separator controls the second gate. The same sequence of events occur as for the prep reading. The third automatic position on the indexing switch presents leaf information to the electronic counter without clock pulses. The counter counts one pulse every time a scanning line passes leaf. In this manner the relative size of particles is determined. For example, a large leaf of 10% total area will give a relatively low count depending upon the number of scans crossed. A 10% leaf area consisting of many small particles gives a much higher count. Finite standards can be established on this basis. This is explained in Figure 1, where the leaf area is diagramed in relation to the full count.

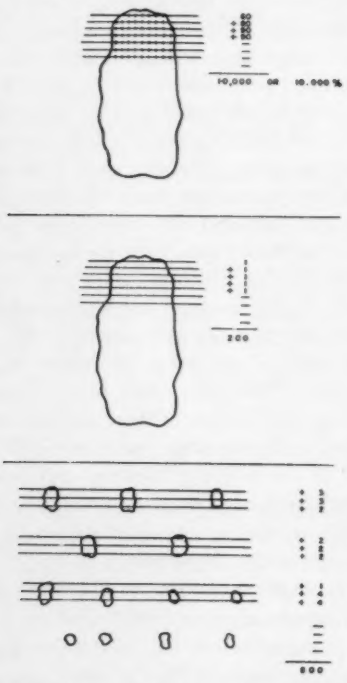

Fig. 1. The relation of leaf size to count. (Top) pulse bunches extend across the width of the trash particle; (middle) only single scanning recorded; (bottom) same area as above, but small particles yield a higher count.

The automatic fourth step consists of the reflectance reading only on the tristimulus coordinate. This is in percentage reflectance as compared against magnesium oxide as a standard 97.5%. This information, which is in voltage form, actuates an analog-to-digital converter to change it to a form acceptable to the printout device. Position five gives the same results in the yellow range.

The complete procedure for reading and recording the five pieces of information requires approximately 5 sec.

Figure 2 is a front view of the Cotton Classer. The low cabinet provides a tabletop surface in which is centered the viewing window of the apparatus. A bale sample of cotton is being held in place over the window by a weighted block. To the rear of the sample there can be seen a pair of dial gauges which record the yellowness and reflectance of the cotton. At the top of the taller cabinet is a panel containing the monitor screen of the television circuit; the sample being surveyed is pictured on the screen. Di-

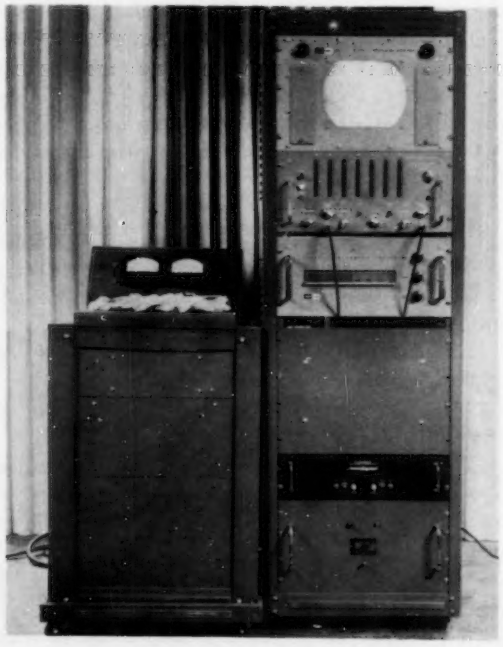

Fig. 2. Front view of the Cotton Classer.

rectly below the monitor screen the seven-place readout counter can be seen.

Figure 3 is a closeup of the operator's table. The viewing window is in the center foreground. The meters for reflectance and yellowness are panel mounted directly behind the window. To the side of the viewing window is a row of signal lights fronted by the control button. The sequence of readings may be noted on the signal lights as preparation, leaf, and leaf count. The last signal is a "ready" light to be used during the warmup operation.

The arrangement of parts in this pilot model may be seen in Figure 4, which is a rear view of the instrument with the cover plate removed from the larger cabinet.

A block diagram of the instrument (Figure 5) shows the relationship of these parts in the total assembly.

Experimental Procedure

The complexities of cotton classing were explained in the Introduction, but as Cox [2] states, "Classing is concerned primarily with matching or identifying samples of cotton by comparing them with other samples set up as types or standards." The reference standards are supplied by the Federal Government through the United States Department of Agriculture and were used as the basis for a study of the operation of the Cotton Classer.

Since the Cotton Classer does not disturb or disrupt the sample in any way, it was possible to read and reread a given sample any number of times to gauge the reliability and duplicability of the findings. Some work was also done on bale samples to discover the uniformity of a number of breaks within a sample.

The data were obtained on six grade boxes (1954 grades), each grade box containing five or six stratified samples within its grade. The data included the following:

	Replications on each stratified sample
Leaf area	4
Leaf count	3
Preparation	3
Reflectance	0
Yellowness	0

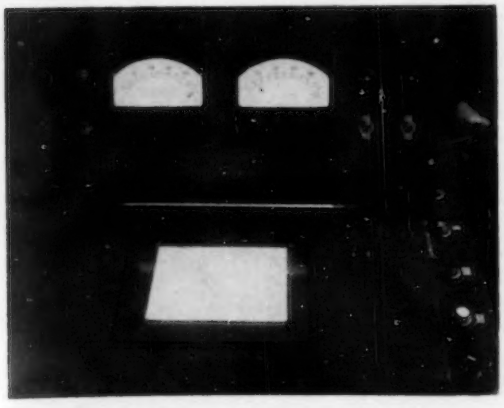

Fig. 3. Closeup of the operator's table.

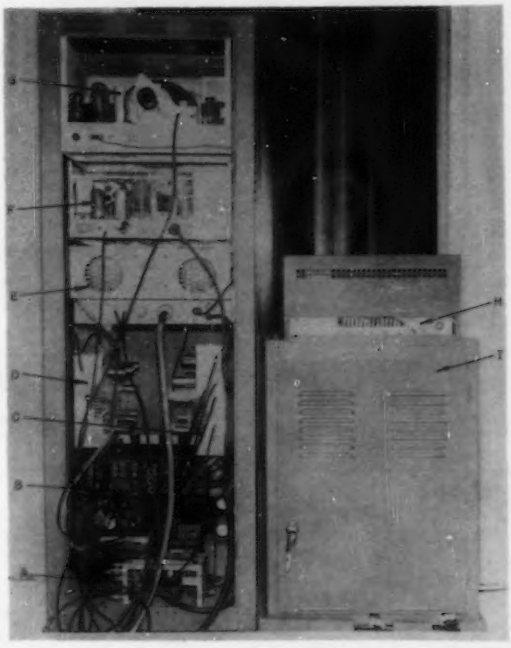

Fig. 4. Rear view of the Cotton Classer with cover plate removed. (A) Selector (operated from the main control button); (B) power supply regulator; (C) master clock; (D) separator; (E) camera control; (F) counter; (G) monitor; (H) colorimeter enclosure; (I) camera housing.

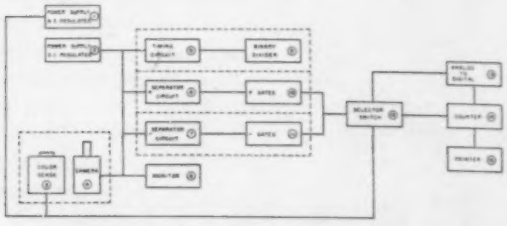

Fig. 5. Block diagram of the Cotton Classer.

Tables I–III summarize the statistical analysis of the data.

With respect to distinguishing among grades, 14 out of 15 possible comparisons based upon leaf area and 14 out of 15 comparisons based upon leaf count are statistically significant. This indicates that the Cotton Classer does distinguish the different grades represented by these samples.

With respect to distinguishing among grades based on preparation, the Cotton Classer findings do not satisfactorily fit the grading system.

An analysis of variance was made on each grade box to determine whether or not the estimated total variance within the grade was significantly greater than the estimated variance within the stratified samples. In all cases for leaf, leaf count, and prep, the variance within the stratified samples was significantly lower than the estimated variance among stratified samples (within grade) for all of the six grades tested. This is a generalized finding. Due to the fact that the proper order for each stratified sample within grades was not known, no definite statement can be made from this data with respect to the Cotton Classer distinguishing among specific stratified samples for any grade.

While a thorough statistical treatment was made of the available data, a number of factors were not included in the experimental design of checking out this instrument due to the very short time available for the study. Therefore, from a statistical viewpoint, the long range stability of the above data is subject to further consideration as the work progresses.

Having establishing statistically the basis for further comparisons of data, some unclassified cottons were examined and rated against the grade box data. Table IV shows the results obtained on five typical samples from a lot of fifty tested.

TABLE I. Average Values for Leaf Area, Leaf Count, and Prep for Six Grade Boxes (Cotton Classer Data)

	Leaf area	Leaf count	Prep
GB 11 MSP	12.1	8.0	17.5
GB 14 SLMS	12.5	8.7	18.4
GB 13 SGO	13.5	10.9	15.3
GB 9 SM	11.7	7.9	18.6
GB 5 GO	14.6	8.9	17.7
GB 6	11.8	10.0	20.1

TABLE II. Comparison of Classification Data (Cotton Classer)

Significance of Differences Among Grades Based on Cotton Classer Data

	GB 11 MSP	GB 14 SLMS	GB 13 SGO	GB 9 SM	GB 5 GO	GB 6
Leaf						
GB 11 MSP	—	HS	HS	HS	HS	HS
GB 14 SLMS	HS*	—	HS	HS	HS	HS
GB 13 SGO	HS	HS	—	HS	HS	HS
GB 9 SM	HS	HS	HS	—	HS	NS
GB 5 GO	HS	HS	HS	HS	—	HS
GB 6	HS	HS	HS	NS†	HS	—
Prep						
GB 11 MSP	—	NS	HS	S	NS	HS
GB 14 SLMS	NS	—	HS	NS	NS	HS
GB 13 SGO	HS	HS	—	HS	HS	HS
GB 9 SM	S‡	NS	HS	—	NS	HS
GB 5 GO	NS	NS	HS	NS	—	HS
GB 6	HS	HS	HS	HS	HS	—
Leaf count						
GB 11 MSP	—	HS	HS	S	HS	HS
GB 14 SLMS	HS	—	HS	HS	NS	HS
GB 13 SGO	HS	HS	—	HS	HS	HS
GB 9 SM	S	HS	HS	—	HS	HS
GB 5 GO	HS	NS	HS	HS	—	HS
GB 6	HS	HS	HS	HS	HS	—

* HS—Highly significant; significant at both 5% and 1% (statistically).
† NS—Not significant at 5% or 1% (statistically).
‡ S—Significant at 5% but not at 1% (statistically).

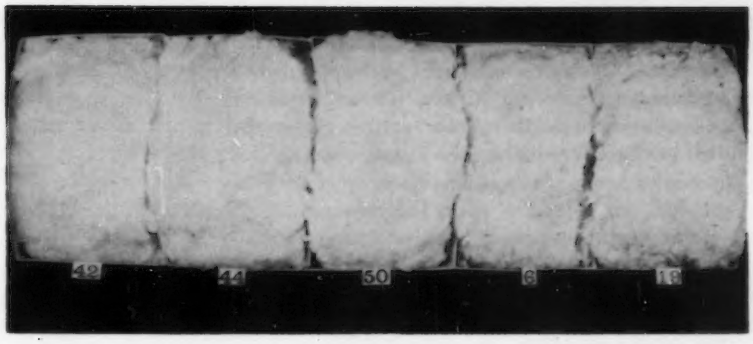

Fig. 6. Cotton samples evaluated in Table IV.

TABLE III. Analysis of Variance Within Classifications

Significance of Variance Differences Within Grades

	Leaf	Prep	Leaf count
GB 11 MSP GB 14 SLMS GB 13 SGO GB 9 SM GB 5 GO GB 6	Significant at 1% level		

TABLE IV. Typical Data on Five Cottons *

Sample no.	Reflectance	Yellow	Leaf	Prep	Machine call
6	71	23	4.50	12.4	SGO–GO and yellow
19	69	37	3.81	14.5	Leafy–yellow–below grade
42	74	23	2.90	12.0	M–yellow
44	82	20	2.87	10.8	SM
50	75	23	2.93	8.6	M

* These specimens are shown in Figure 6.

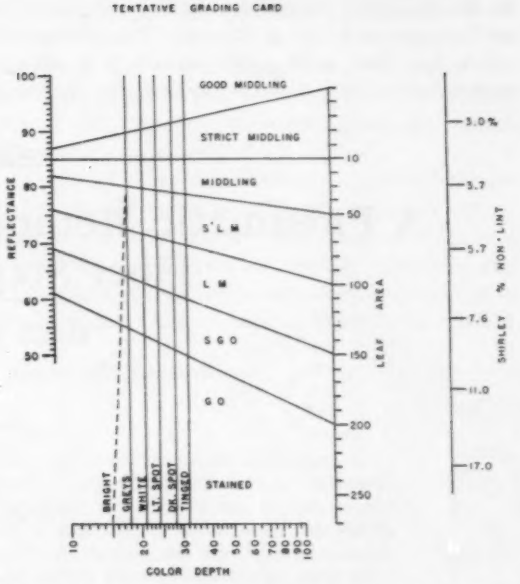

Fig. 7. Nomograph based on cotton grade boxes for use with the Cotton Classer.

The grades placed on the cotton specimens were established from the original data taken from the grade boxes. An assembly of the usable data provided by the Cotton Classer using the grade boxes as a standard makes possible derivation of a nomograph shown in Figure 7. It is to be understood that newer grade boxes are in use than those upon which this particular nomograph was based. A modernization of the nomograph data is underway at this writing.

Conclusions

An apparatus has been described which will provide quantitative values of five visual parameters useful in classing cotton. The findings obtained on the experimental model are shown to be statistically reliable.

Methods have been evolved to render the data suitable for classing cotton in accordance with a specific set of grade boxes. Some of the data obtained, however, are not necessary for the classing operation but serve as supplementary information. These latter parameters are preparation and number of leaf particles. The elements of leaf area, yellowness, and reflectance appear adequate to classify cotton in accordance with current methods.

Through use of a nomograph the data obtained on the Cotton Classer were used to classify a number of

unknown cottons, some of which are illustrated in the text of this paper.

Preparation, which is measured as the area lighter than background on the Cotton Classer, does not appear to weigh into the classer's call to any extent but is readily measured on the Classer. The particle size measurement which is obtainable on the instrument does not appear to affect the classer's call directly, although it is known to be increasingly important in the evaluation of pepper type trash and is associated with what has been called overworked cotton.

Details of a simplified cotton classing system based on the use of the instrumental findings remain to be worked out with the apparatus. The nomograph which has been used experimentally will require modernization for use in conjunction with the latest set of grade boxes and will utilize three points of a five-point measuring system.

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A Pneumatic Method of Measuring Cotton Fiber Staple Length[1]

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Abstract

A pneumatic device for measuring the mean and upper-half mean fiber lengths is described. Air is drawn through a cotton fiber beard (formed with two combs) placed over a narrow slit to form the fourth arm of a pneumatic Wheatstone bridge. It is shown that the unbalanced deflection of the "pneumatic galvanometer" across the bridge varies correctly with the number of fibers over it to enable measurement of the mean value from curves automatically drawn by the device.

For seventeen cottons data are presented which compare the results of this method with those obtained with the Fibrograph.

Introduction

Since the pneumatic bridge developed for the Roving Evener (reported at the Cotton Research Clinic last year) would so easily measure the size of sliver or roving, it was thought the same bridge arrangement might be used to give readings proportional to the number of fibers in a beard at any distance from the base. The beard is formed by the use of two combs, as is done for the well known Fibrograph developed by Hertel [1, 2]. Air is drawn through the beard through a thin 0.007 × 5.0 in. slit in the wall of a manifold placed at right angles to the fibers. Figure 1 shows a schematic diagram of the device. It is seen that the cotton and the slit form the fourth resistance in a pneumatic Wheatstone bridge. Corresponding to the galvanometer of the electrical bridge, a differential bellows is used to measure the amount of unbalance caused by the cotton covering the slit and also to move the pen, automatically recording the degree of unbalance on a chart as the cotton beard is drawn over the slit from base to tip.

[1] Presented at the Cotton Research Clinic of the National Cotton Council of America, Pinehurst, N. C., February 13, 1958.

The bridge is operated unbalanced with a vacuum approximately equivalent to a 12-in. head of water; for the thickest beard, the differential pressure across the bridge may approximate 2 in. of water. Of course, a higher vacuum would give greater sensitivity. The resistances R_1 and R_2 are usually less than half R_3 and R_x, but are not critical, being made of 1 or 2 in. of tubing 0.11 in. inside diameter.

The 8-in. pen arm is mounted on a shaft that is caused to rotate by the differential pressure across the bellows. The pen rests lightly on the card in a holder which is moved under the pen by the same sliding frame that carries the fiber comb.

By slowly moving the frame, the cotton beard is drawn past the slit and simultaneously the card is moved under the pen so that a curve is drawn. The mean and upper-half mean fiber lengths are measured by drawing tangents to the curve, as is done with the Fibrograph. This procedure is valid if the distribution of fiber lengths in the beard is similar to that obtained by the sliver clamping method [3] in which the beard is made by clamping a sliver at a point and combing away the loose fibers on one side of the clamp.

Discussion of the Problem

For this pneumatic method to work, the deflection of the pen should be proportional to the number of fibers across the slit. Whether or not this is true, as would be expected, depends on several things that affect the galvanometer response to change in the resistance R_x and on the way the resistance of the slit changes with the number of fibers over it. For the Wheatstone bridge it can be shown that when R_3 is two or more times greater than R_1 and R_2, the differential pressure across the bridge rises almost linearly with the increase in the resistance R_x. Assuming the resistance of the slit to be inversely proportional to its area, and that the galvanometer deflection is proportional to the increase in the slit resistance, the deflection caused by covering the slit should increase faster than the percentage of the slit covered.

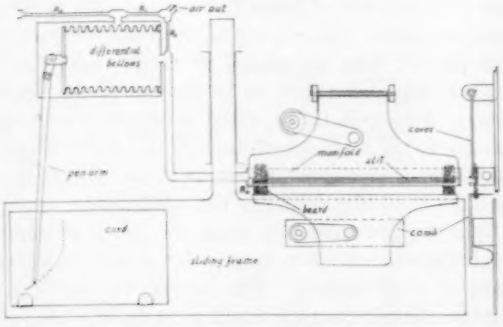

Fig. 1. Pneumatic staple length meter.

The theoretical increase in resistance caused by progressively covering the greater proportions of the slit (with tape) is shown in Figure 2. The deflections are plotted to a proper scale to make the two curves fit as closely as possible. It is seen that the agreement is good for covering approximately 70% of the slit, which usually exceeds the working range in normal use of the instrument. The agreement of the curves shows that total effective resistance of R_x is the slit itself, which is not much affected by the resistance of the connecting tube. Trying several sizes of tubing for this connector does not seem to change the shape of the curves given for various fiber beards.

From the above considerations it is noted the deflection is not linear with the percentage of slit actually sealed off. Therefore, if equal increments in the number of fibers over the slit effectively seal equal increments of the slit, the deflection can not be proportional to the number of fibers across the slit.

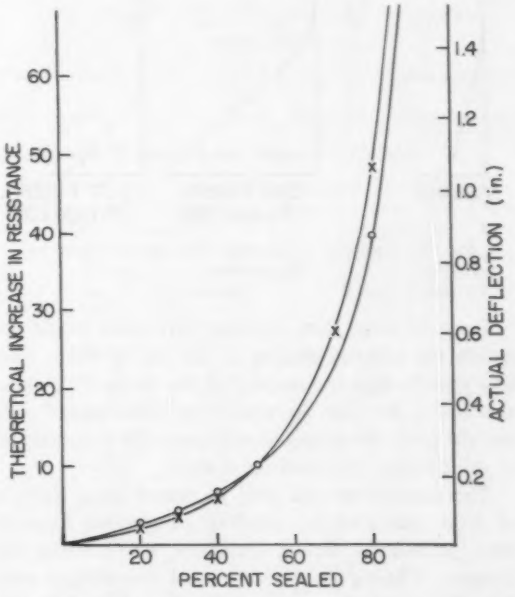

Fig. 2. Theoretical increase in resistance and actual deflection vs. percent of slit sealed. ○ = resistance; × = deflection.

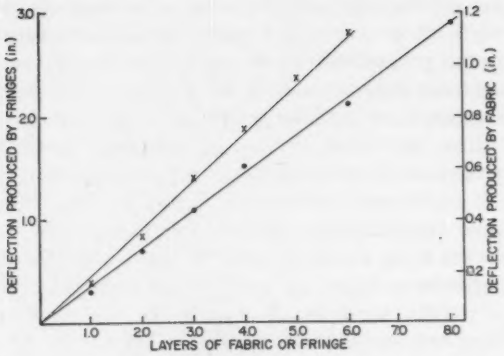

Fig. 3. Galvanometer deflection by layers of cotton fabric and nylon fiber fringe. ○ = fabric layers; × = fringe layers.

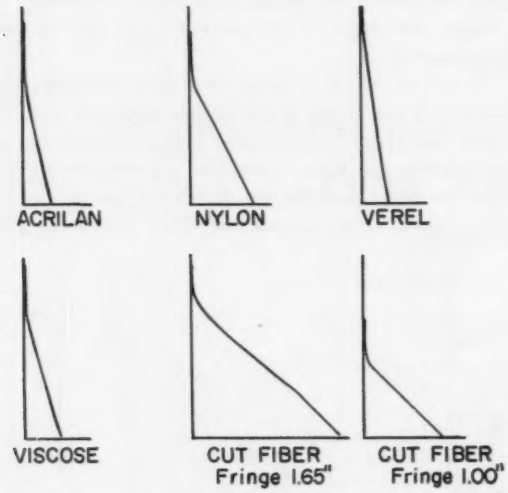

Fig. 4. Tracings of straight line curves drawn by the machine.

It would seem, then, that the instrument would fail unless the effective sealing of the slit by fibers rises less rapidly than the number of the fibers. To show that this is the case, layers of 1-oz. fabric placed over the slit give deflections almost perfectly proportional to the number of layers up to eight.

The instrument can also be tested using fringes of fiber prepared by winding continuous filament fiber on boards, taping crosswise, and cutting the fringes. Placing successively equal fiber fringes over the slit again gives deflections proportional to the number of fibers across the slit, proving that the sealing efficiency of fibers is less as more fibers are laid over the slit. Figure 3 shows the deflections given by layers of fabric or fibers.

If a fiber with a single staple length is used in the sliver clamping technique, the beard produced should have a linear increase in fibers per cross section from the tip to the base of the beard. Assuming that preparing the beards with combs gives the same fiber distribution, the instrument should draw a straight line for such beards. For some manmade fibers the instrument gives practically straight lines.

By cutting fiber fringes in triangular shapes, one can be sure of having a strictly linear increase in fibers from tip to base of the fringe. For such fringes made of nylon fiber, the curves are practically straight lines. Figure 4 shows tracings of curves drawn by the instrument for Acrilan, nylon, viscose, and Verel staple and for nylon cut fringe.

The straightness of these lines and the fact that their slope is correct to give their staple lengths seems to prove that the device does indeed measure the number of fibers at each point along the beard and that, for single staple lengths, preparing beards with combs gives the theoretically correct linear distribution of fiber ends in the beard. It seems surprising that the sealing efficiency for added fibers decreases at just the right rate to make the galvanometer deflection proportional, over a wide range, to the number of fibers across the slit.

Some synthetic fibers, especially when using thick beards, give curves convex upward. This must be due to the heavy crimp in the fiber, which is straightened by the cover plate more effectively nearer the tip of the beard than at the base.

Triangular fringes of filament cut to different lengths can be overlapped to check whether or not the machine gives the correct mean length for known distributions. Using two triangular nylon fringes of the same number of fibers 1.70 in. and 1.00 in. long, the instrument draws a curve, with one slope, until the shorter fiber has passed off the slit and with a higher slope from then on to the end of the longer fiber. A tangent to the initial slope gives, with fair accuracy, the mean length as 1.30 in., and a tangent to the final slope gives the length of the longer staple as 1.73 in., which is pretty fair agreement.

For the instrument to work for cotton it should give the correct mean lengths for a wide distribution of fiber lengths. The curves for cotton are always convex downward and seem to have the correct shape for the usual tangent lines to indicate both

the mean and upper-half mean lengths. Seventeen cottons supplied by the U.S.D.A. and A.C.M.I. laboratories at Clemson were tested. These were check cottons for which the Fibrograph lengths were supplied. Table I gives the results of the pneumatic method compared with the given Fibrograph values. On each of the 17 cottons, 6–9 readings were taken. (This first apparatus uses a single comb, so the average for both combs is considered one reading for each sample.) Though it may be customary in routine testing to discard readings differing widely from the mean, in this case the values in the table include the results of every test taken on each cotton and for every cotton tested.

In Figures 5 and 6 are shown the values for mean and upper-half mean length plotted against the Fibrograph readings for each cotton. It should be realized that the deviations of individual results from the general trend can be due either to errors of the Fibrograph or of this method or of both. Incidentally, the consistency of the results may increase confidence in the reliability of the Fibrograph.

Conclusions

It is noted that the mean length values are slightly on the low side. Possibly part of the reason for this is that the air pressure in this work, not being perfectly controlled, falls from 0.25 to 0.5 in. in 12 in. as the beard is moved off the slit. Keeping the pressure constant should make the curve slightly less convex downward and therefore give slightly higher values for the mean length but have relatively little effect on the upper-half mean values.

The apparatus seems not to be very critical as to beard thickness. None of the samples was weighed, and for half of the readings different beard thicknesses were purposely used.

The method does not require any certain air pressure but only that it remain fairly constant during the

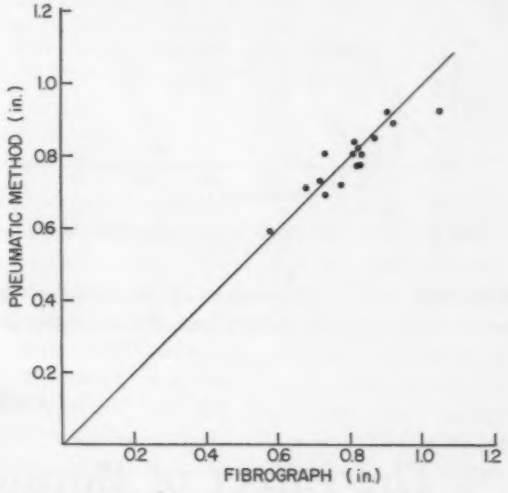

Fig. 5. Mean length; pneumatic method vs. Fibrograph.

TABLE I. Comparison of Mean and Upper Half Mean Lengths by Fibrograph and Pneumatic Method

Mean length, in.				Upper half mean length, in.			
Length		Accuracy		Length		Accuracy	
Fibrograph	Pneumatic	Range	Standard deviation	Fibrograph	Pneumatic	Range	Standard deviation
.73	.74	.14	.04	.58	.60	.15	.02
.87	.90	. 5	.07	.68	.72	.16	.05
.89	.97	.13	.07	.73	.81	.20	.06
.90	.94	.13	.03	.72	.73	.19	.06
.91	.94	.13	.04	.73	.71	.12	.04
.98	1.03	. 9	.03	.82	.83	. 6	.02
.99	1.02	.22	.05	.78	.72	.10	.04
1.00	1.02	. 4	.02	.82	.77	. 8	.03
1.04	1.10	. 9	.03	.82	.82	. 5	.02
1.05	1.04	. 9	.03	.83	.77	.14	.04
1.06	1.06	.13	.05	.84	.80	. 8	.03
1.06	1.09	.10	.04	.81	.80	.11	.04
1.11	1.15	. 6	.02	.87	.85	. 7	.02
1.15	1.16	. 7	.03	.92	.89	.14	.05
1.21	1.24	.16	.06	.91	.92	.22	.10
1.31	1.32	.11	.03	1.01	1.01	.12	.05
1.36	1.33	. 9	.03	1.04	.92	.14	.04

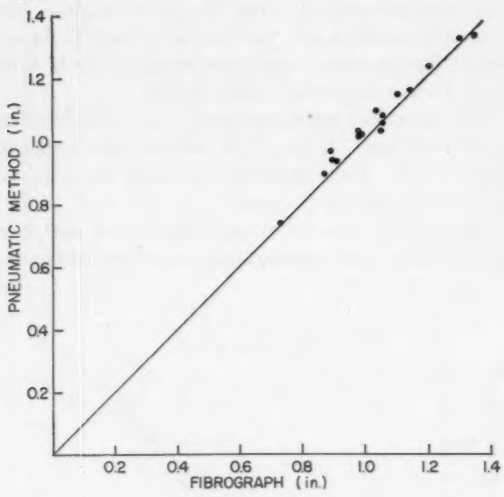

Fig. 6. Upper-half mean length; pneumatic method vs. Fibrograph.

individual tests. The pressure of the cover on the beard is not critical, though probably improved results might be obtained by having a better control of it.

Arranging a small motor to move the comb at a constant rate may give better results than these obtained by moving the comb by hand. Possibly using the galvanometer balanced as a null instrument might improve the results. This can be done by using a second slit in the arm R_3 mounted at right angles to the slit for the cotton. A wedge type shutter over the second slit would be moved horizontally with the card while the pen would be lowered vertically with the cotton comb. There is a question whether the improvement would justify the additional cost and complication in operation.

Development of this instrument is still in progress, but results to date seem to show some promise for a pneumatic method of measuring staple length.

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The Effect of Bottom Front Roll Run-Out in Spinning on Yarn Quality and Processing Performance[1]

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Abstract

Measurement of bottom front roll run-out at every spinning stand position in a typical mill indicates that only a small percentage of the stands have run-outs that are excessively high. It was found also that square neck jointed rolls in the two plants studied have higher roll run-outs than screw neck jointed rolls. A reasonable explanation for this is that the square neck jointed rolls generally receive rougher handling, since considerable force is required when separating or putting together the sections of the rolls on the spinning frame.

Periodic irregularities are introduced in the yarn by eccentric bottom front rolls which lower the quality of the yarn being produced. With increases of bottom front roll run-out from 0.000 in. to 0.010 in., the following properties of 31/1 warp yarn were affected to the extent shown:

Break factor	11% decrease
Single end strength (oz.)	15% decrease
Single end elongation	14% decrease
Percent nonuniformity (% NU)	12% increase

As the roll run-out increased, the pattern on yarn appearance boards became more distinct. Analysis of the yarn properties indicated that yarn quality was appreciably lowered at bottom front roll run-outs in excess of 0.0055 in.

A study of the processing performance of the yarn produced at various bottom front roll run-outs indicates that yarns produced at high roll run-out positions are more difficult to process in spooling and weaving.

In this study, there was an increase of 12% in the number of breaks in spooling as the bottom front roll run-out increased from ranges of 0.000–0.003–0.006 in. and higher. With these same ranges of roll run-outs, warp stops in weaving increased 48% when the weavability of the yarn produced at low roll run-out positions was compared with yarn produced at high roll run-out positions in spinning.

The appearance grade of 80 × 80 print cloth, woven with filling yarns produced at various roll run-out positions, was found to decrease with increases in bottom front roll run-outs. All graders that ranked the fabrics selected the fabric having the highest roll run-out as being the worst fabric in the group. However, no correlation was found between the appearance grade of 110 × 58 broadcloth woven with warp yarn produced at different bottom front roll run-outs in spinning.

Introduction

With the introduction of spinning systems employing relatively high drafts and high production rates, manufacturers of high quality yarns and subsequent fabrics found that machine adjustments and tolerances required more careful attention in order to maintain a high level of quality. One such critical tolerance has been found to be the run-out, or eccentricity, of drafting rolls.

Eccentric rolls produce yarn irregularities either by movement of the nip of the rolls or by varying surface speed of the rolls. Eccentric bottom rolls which are positively driven cause varying surface speeds as well as movement of the nip. However, since top rolls are surface driven, only movement of the nip is present with defective top rolls. With a constant amount of stock being fed to an eccentric bottom roll and a varying rate of delivery due to changes in surface speed, thick and thin places are produced as a result of periodic increases and decreases in draft (i.e., draft = surface speed delivered/surface speed fed). Movement of the nip of the roll, caused either by top or bottom eccentric rolls, introduces irregularities due to variations in draft and variation in the settings between the rolls.

Gregory and Tyson [1] state that the additional variation introduced in the yarn by eccentric rolls is proportional to eccentricity × draft/roll circumference, with the draft being the draft in the zone just prior to the eccentric roll. For this reason, roll run-out becomes of increasing importance on higher drafting systems; since higher drafting systems utilize higher front zone draft, run-out of bottom front roll becomes extremely important.

At the 1957 Cotton Research Clinic sponsored by the National Cotton Council of America, Dougherty [2] reported that data from pilot plant studies showed that bottom front roll run-out of the spinning frame affects yarn quality more noticeably than run-out of the bottom middle or bottom back rolls. Finer count yarns were also affected more than coarse yarns at the run-out of the bottom front roll.

The pilot plant data were of considerable interest, but a number of questions pertaining to yarn manufacture by mills interested in this problem remained unanswered. Some of the more important of these questions were: (1) What run-outs actually exist in a typical mill which has not previously attempted to control run-out in spinning; (2) How are the properties of yarn produced under mill conditions affected by bottom front roll run-out in spinning; (3) What is the effect of roll run-out in subsequent processes such as spooling and weaving; and (4) What is the effect of bottom front roll run-out on fabric appearance?

As part of the student research program of the Institute of Textile Technology, a cooperative research project was undertaken in the summer of 1957 between the Institute and a member mill with the objective of obtaining the answers to the questions enumerated.

[1] Presented at the Cotton Research Clinic of the National Cotton Council of America, Pinehurst, N. C., February 12, 1958.

[2] Candidate for M.S. degree.

Measurement of Bottom Front Roll Run-Out in Spinning

Because of vibration on the gauge, accurate measurements of bottom front roll run-out could not be obtained when the frame was running at normal production speeds. Therefore, a method of measuring run-out was devised so that the bottom front roll could be turned very slowly. After disengaging the intermediate draft gear, the front roll gear was replaced by a special crank. By turning the front roll in this manner, measurements could be made very easily. All measurements were made with a standard dial gauge with the magnetic base in the position shown in Figure 1.

Although measuring the roll at every spindle position in the plant would have been desirable, the time allotted to this project prohibited measuring all of the approximately 50,000 spindles in the plant. It was found in this plant that the variation in run-out among spindle positions within a stand was very low, in the neighborhood of 0.002 in. The variation among stands within a frame, however, was extremely large. Measurement of run-out on the fluted boss of the roll at a position near the center of the stand seemed to characterize the run-outs present in that particular stand. Therefore, one measurement was made at every stand in the plant.

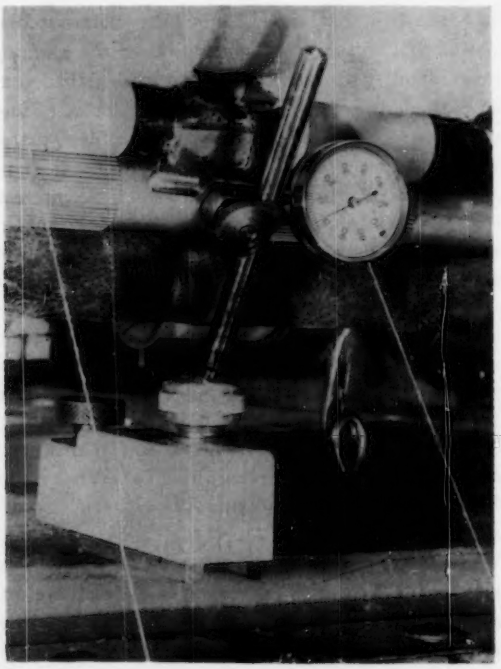

Fig. 1. Dial gauge and magnetic base in the position for measuring bottom front roll run-out.

Two teams were used in order to increase the speed at which the measurements were made. The first team consisted of one man to turn the front roll slowly with the crank and a second man to make the measurements along the front roll. The second team consisted of two spinners to piece up all the ends and to put the frame back into production as rapidly as possible. In an 8-hr. day, these two teams were able to measure 16–18 frames.

Results

In Plant A, 7867 stands were measured for bottom front roll run-out. Of these, 7447 stands were on frames having screw neck jointed rolls and 420 were on frames having square neck jointed rolls. In a survey of another plant, Plant B, in the same organization, 1380 stands of square neck jointed rolls and 1224 stands of screw neck jointed rolls were measured. The distributions of these stands at the various run-outs are shown in Figures 2 and 3.

Most of the stands on frames having screw neck jointed rolls have relatively low run-outs, only 3% of the stands being 0.005 in. and higher in Plant A and 1% of the stands in Plant B being 0.005 in. and higher. The frames having square neck jointed rolls in these two plants have a higher percentage of the stands at higher run-outs. Forty percent of the

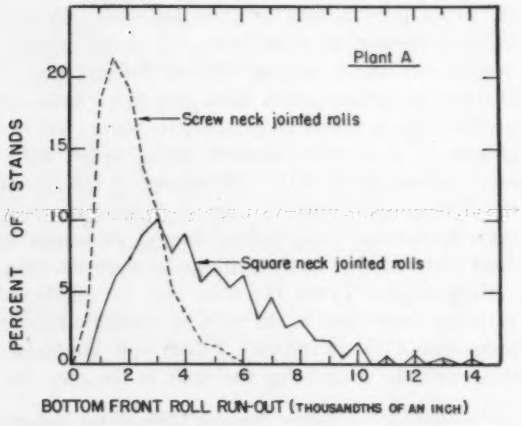

Fig. 2. Distribution of stands of screw neck jointed rolls and square neck jointed rolls at the various bottom front roll run-outs in Plant A.

stands in Plant A and 16% of the stands in Plant B have run-outs 0.005 in. and higher.

It is the consensus in the industry that square neck jointed rolls generally will have higher run-outs than screw neck jointed rolls. Although this is true in the two mills studied, it cannot be concluded that these rolls are more defective when produced by the manufacturers. A mill may receive a perfectly true roll from the roller manufacturer, but when it is put in production, excessive run-outs may be present. Improper handling of rolls during cleaning, worn stands, improperly aligned stands, and sagging frames are only a few faults at the spinning frame which may increase the run-out of the roll. It is true, however, that a mill cannot generally obtain run-out lower than that produced by the manufacturer. Square neck jointed rolls usually have to be forced together, or separated, and probably this rougher handling of the rolls is the factor which makes square neck jointed rolls have higher run-outs.

Influence of Bottom Front Roll Run-Out on Yarn Properties

To study the effect of bottom front roll run-out on yarn properties, 31/1 warp yarn was produced because it represents the average warp count in the plant and also the greatest bulk of the warp production. For purposes of yarn quality studies, the classifications of bottom front roll run-outs used are shown in Table I.

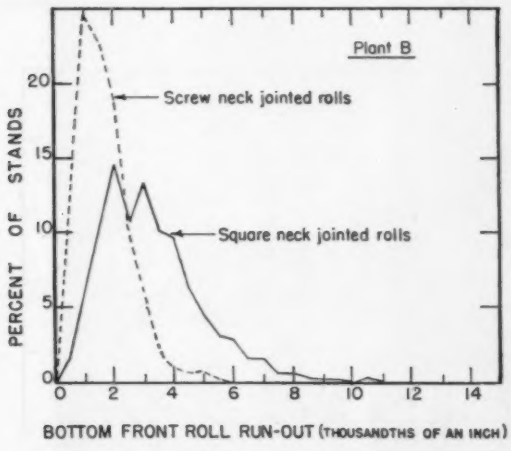

Fig. 3. Distribution of stands of screw neck jointed rolls and square neck jointed rolls at the various bottom front roll run-outs in Plant B.

Eight bobbins were used for each test in each group. These bobbins were selected randomly from stands having the various run-outs. All yarn tested for yarn quality was produced at the spindle position that was measured for the run-out of the stand. The properties studied were break factor, single end strength, percent elongation, percent nonuniformity, and yarn appearance.

Break factor was determined on the Scott-J Tester using three skeins per bobbin. Single end strength was determined with the Suter Single Strand Strength and Elasticity Tester with Oil Plunger Controlled Pull. This tester has a gauge length of 18 in. and is timed so that the carriage moves from top to bottom in 18 sec. Twenty breaks were made per bobbin. Both the strength and elongation were recorded for each break.

Per Cent Nonuniformity was determined on the Brush Uniformity Analyzer set at a 10-ft. sensitive length and a speed of 5 ft./sec. Readings were made every 10 sec. on the Automatic Evaluator. A minimum of two tests of 1000 ft. each were made on

TABLE I. Classification of Bottom Front Roll Run-Out Used in Yarn Quality Evaluation Study

Group	Range of roll run-outs, in.
A	0.000–0.0015
B	0.002–0.0035
C	0.004–0.0055
D	0.006–0.0075
E	0.008 and higher

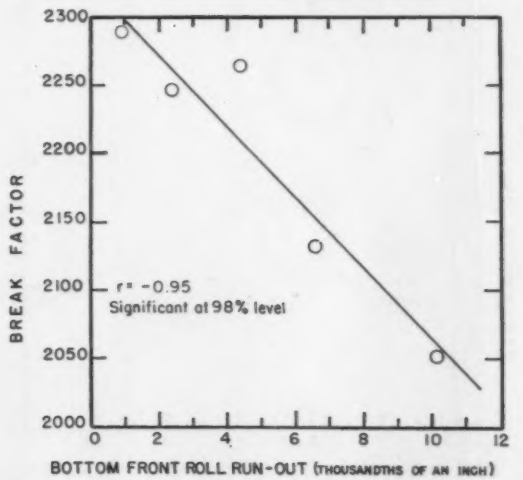

Fig. 4. Influence of bottom front roll run-out on break factor of 31/1 warp yarn.

each bobbin. Yarn appearance boards were prepared on $9\frac{1}{4} \times 5$ in. boards. A length of $9\frac{1}{4}$ in. was found to be suitable for detecting periods produced by 1-in. diameter front rolls.

Results

The influence of bottom front roll run-out on the various yarn properties is shown graphically in Figures 4, 5, 6, and 7. With increases of bottom front roll run-outs from 0.000 in. to 0.010 in., the quality of the yarn produced decreased, as shown in Table II.

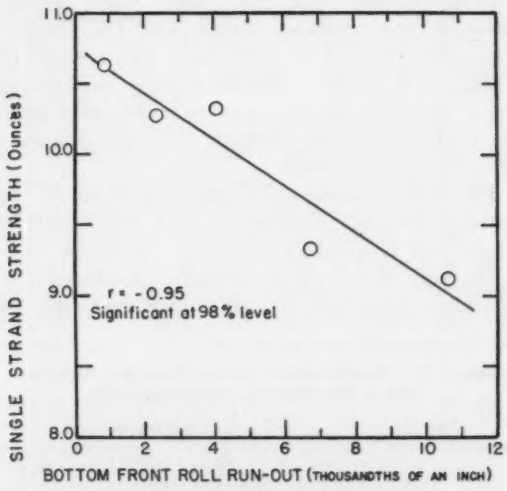

Fig. 5. Influence of bottom front roll run-out on single strand strength of 31/1 warp yarn.

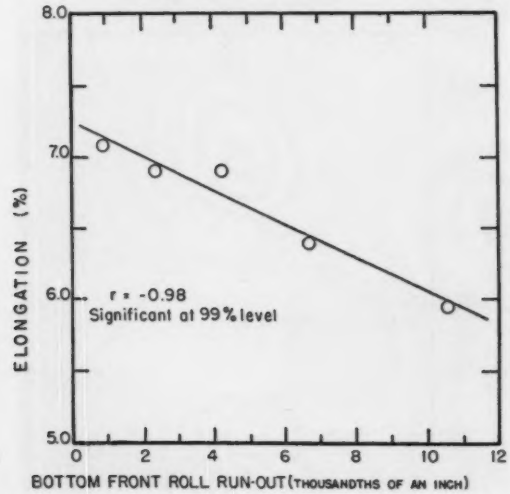

Fig. 6. Influence of bottom front roll run-out on single end elongation of 31/1 warp yarn.

As the roll run-out increased, the pattern on the yarn appearance board became more distinct. Photographs of three typical yarn boards are shown in Figure 8. The yarns shown from left to right were produced at 0.001, 0.005, and 0.0095 in. roll run-outs, respectively. The pattern on the board made with yarn from a position having 0.0095 in. roll run-out is very distinctive and shows a period of 3.1416 (1% contraction).

Analysis of the yarn properties indicated that yarn quality of 31/1 warp was lowered appreciably at bottom front roll run-outs in excess of 0.0055 in. In order to eliminate stands producing poor quality because of roll run-out, it was recommended that all stands having 0.005 in. and higher run-outs be corrected. Reworking of rolls having high run-outs allowed the mill to lower the run-out to a safe level.

The upper limit may vary slightly from plant to plant because of different drafting systems, draft employed, diameter of rolls, and the yarn count spun. Individual mills interested in this problem should study yarn quality at the various roll run-outs to determine acceptable tolerances.

Influence of Bottom Front Roll Run-Out on Processing Performance

Processing performance was studied in spooling and weaving using three types of warps classified according to bottom front roll run-out. The classifications of roll run-outs used in these studies are given in Table III.

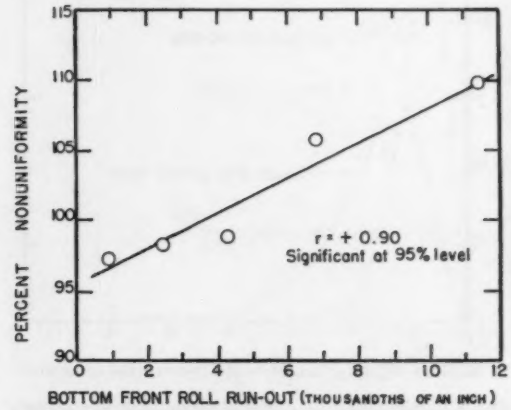

Fig. 7. Influence of bottom front roll run-out on nonuniformity (% NU) of 31/1 warp yarn.

The stands were color coded into the various types of warps. After segregation of bobbins by stand run-out, the yarn was processed into three types of warps. The yarn was first processed on the same side of a Barber-Colman "C" type spooler using the normal settings, speeds, and conditions for 31/1 warp. Yarn breaks were recorded, except in clear cases of yarn tangling where only a yard or so of yarn was left on the bobbin. A minimum of 5000 bobbins was studied for each type of warp.

Using the cheeses produced in the spooling studies, each type of warp was made into nine section beams of 420 ends and two section beams of 325 ends, each beam being 5200 yards in length. The same conditions of slashing were used in producing five loom beams of 1000 yards each of each of the three types of warps.

The loom beams were then tied in on Draper Model-E looms making a 40-in., 110 × 58, 3.33 broadcloth. Warp stops on these warps were observed for 1440 loom hours, which was enough to give a statistically reliable test. A Latin square weaving plan was used to eliminate variables other than differences in the three types of warps. The plan employed is shown in Table IV.

In Table IV each period consisted of 8 hr.; A, B, and C are types of warps classified according to roll run-outs, as described in Table III.

Results

Yarns produced at the higher roll run-outs in spinning were the most difficult to process in subsequent

TABLE II. Changes in Various Yarn Qualities with Increases in Bottom Front Roll Run-Out of the Spinning Frame from 0.000 in. to 0.010 in.

Yarn property	Range	Approximate change for roll run-out changes from 0.000 in. to 0.010 in., %
Break factor	2289–2052	−11
Single end strength	10.64–9.12 oz.	−15
Single end percent elongation	7.08–5.95	−14
Per Cent Nonuniformity	97.17–109.92	+12

TABLE III. Classification of Bottom Front Roll Run-Outs Used in the Yarn Processing Performance Study

Type warp	Range of run-outs, in.
A	0.000 –0.003
B	0.0035–0.0055
C	0.006 and higher

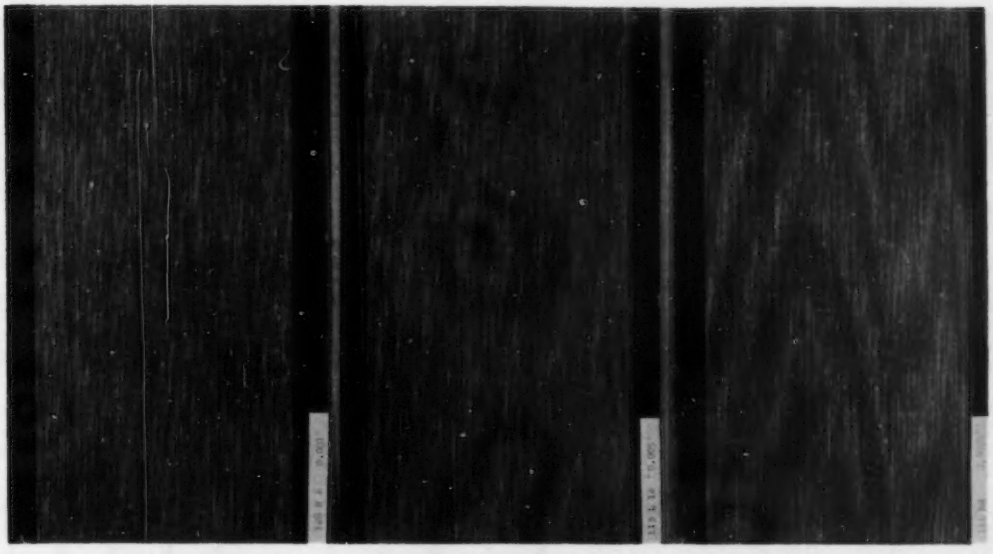

Fig. 8. Yarn appearance boards of 31/1 warp yarn produced at spindle positions having bottom front roll run-outs of 0.001, 0.005, and 0.0095 in. (left to right).

operations, as shown in Table V. At the higher roll run-outs, the number of breaks observed in spooling was the greatest. An increase of approximately 1% in spooling rejects was also found to occur.

It may also be seen from Table V that there is a 12% increase in the number of warp yarn breaks at the highest (Warp C) run-out when compared to yarns made at the lowest run-out (Warp A).

Records were kept of stops in warping; a tendency for more breaks to occur at the highest run-out thus was found. However, not enough breaks in warping were observed to give a statistically reliable test.

In order to make an analysis of the data of warp stops in weaving, only warp stops that could be attributed to roll run-out were considered (*viz.*, weak threads, gouts, and unclassified warp stops). Stops caused by mat-ups, knots, mechanical faults of the loom, etc., were not included in this study. The results obtained in weaving are summarized in Table VI.

Significance of the warp stop averages between Warps A and B grouped together against Warp C showed significance at the 99.9% level. A variance analysis of the warp stop data in which consideration was given to five sources of variation (i.e., individual loom, loom group of five looms each, time period, type warp, and residual or error in the test) also showed a significant difference in the three types of warps at the 99.9% level. All of the other sources of variation were nonsignificant.

Therefore, it can be concluded that warp stops increase as the bottom front roll run-out increases. In this experiment, there were increases of 12% in warp stops between Warp A and Warp B, 32% increase between Warp B and Warp C, and a total increase of 48% between the best warp (A) and the worst warp (C). However, it should be emphasized that these warp stops are only those caused by weak ends, gouts, and unclassified warp stops.

Effect of Bottom Front Roll Run-Out on Fabric Appearance

One sample of cloth made from each type of warp in the weaving test was taken to mill fabric graders to determine if differences in bottom front roll run-out could be detected in the greige goods. The broadcloth samples from Group I were woven with filling yarns of the same run-out and warps of varying run-outs.

Print cloths (80 × 80) also were woven with filling yarns with 0.001, 0.003, 0.004, 0.005, and 0.0065 in. roll run-outs using the same warp (Group II). Graders were asked to rank these fabrics from 1 to 5 in the order of best to worst in appearance.

Results

Analysis of the ranking of the fabrics showed that graders could not detect increasing roll run-outs in the warp direction of the broadcloth (Group I). A possible explanation for this fact is that the relatively tight construction (110 × 58) made the unevenness of the yarn less noticeable. Some areas contained thick and thin areas with a period of the same length as that produced by a 1-in. diameter front roll in spinning.

Roll run-out in the filling of the 80 × 80 print cloth (Group II) was noticeable to the fabric grader. A correlation coefficient of 0.59 was obtained, which

TABLE IV. Latin Square Plan Used in Assessing the Weavability of Types A, B, and C Warps

	Loom number														
Period	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
I	A	A	A	A	A	B	B	B	B	B	C	C	C	C	C
II	A	A	A	A	A	B	B	B	B	B	C	C	C	C	C
III	A	A	A	A	A	B	B	B	B	B	C	C	C	C	C
IV	A	A	A	A	A	B	B	B	B	B	C	C	C	C	C
V	B	B	B	B	B	C	C	C	C	C	A	A	A	A	A
VI	B	B	B	B	B	C	C	C	C	C	A	A	A	A	A
VII	B	B	B	B	B	C	C	C	C	C	A	A	A	A	A
VIII	B	B	B	B	B	C	C	C	C	C	A	A	A	A	A
IX	C	C	C	C	C	A	A	A	A	A	B	B	B	B	B
X	C	C	C	C	C	A	A	A	A	A	B	B	B	B	B
XI	C	C	C	C	C	A	A	A	A	A	B	B	B	B	B
XII	C	C	C	C	C	A	A	A	A	A	B	B	B	B	B

TABLE V. Effect of Bottom Front Roll Run-Out in Spinning on the Number of Breaks and Percentage Rejects in Spooling

Type warp	Range of roll run-outs, in.	Number of breaks observed	Total number of bobbins observed	Spooling rejects, %
A	0.000 –0.003	909	7168	12.68
B	0.0035–0.0055	712	5220	13.64
C	0.006 and higher	739	5220	14.16

TABLE VI. Effect of Bottom Front Roll Run-Out in Spinning on Warp Stops in Weaving

Type warp	Roll run-out ranges, in.	Avg. warp stops/32 loom hr.
A	0.000 –0.003	11.7
B	0.0035–0.0055	13.1
C	0.006 and higher	17.3

is significant at the 99% level. The finer filling (40/1) and the more open construction allowed the defects produced by eccentric bottom front rolls to be seen. Although there was some disagreement among the graders at run-outs from 0.000 in. to 0.005 in., all graders selected the fabric with 0.0065 in. run-out in the filling yarn as the worst fabric in the group.

Conclusions

From this study of bottom front roll run-outs in spinning, the following conclusions can be drawn from the data obtained in the two plants of the mill studied:

1. Square neck jointed rolls have higher roll run-outs than screw neck jointed rolls.

2. Only a relatively small percentage of the stands had excessively high roll run-outs.

3. The quality of 31/1 warp yarn appreciably deteriorates at bottom front roll run-outs in excess of 0.0055 in.

4. As the bottom front roll run-out increases, more rejects in spooling and more warp stops in weaving occur.

5. The appearance grade of 80 × 80 print cloth, woven with various filling yarns, was found to decrease with increase in bottom front roll run-out. On the contrary, no correlation was found between the appearance grade of 110 × 58 broadcloth woven with warp yarns produced at different bottom front roll run-outs in spinning.

Acknowledgments

The assistance of Dr. L. H. Hance, Dr. Jack Compton, Mr. J. E. Dougherty, and Mr. N. L. Enrick of the Institute staff, in planning the experiments and making the arrangements for the conduct of these researches in an Institute Member Mill, as well as the aid given in the selecting of the statistical techniques employed in the analysis of the data and help supplied in the preparation of the manuscript, is gratefully acknowledged. Appreciation is also expressed to Miss Edith Coleman for her assistance in photographical work.

The interest and helpfulness of the management and operating personnel of the Institute Member Mill in which this test was run is recognized as being an indispensable and vital part of this cooperative project. The permission of the management of the mill to release this information is also acknowledged.

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Letters to the Editor

Short communications in the form of Letters to the Editor are intended to provide prompt publication of significant new research results and to permit an exchange of views on papers previously published in the JOURNAL. These communications are not submitted to formal review as are research papers, and the editors do not assume any share of the author's responsibility for the information given or the opinions expressed. When work previously published in the JOURNAL is the subject of critical comment, the authors of the original paper are given an opportunity to submit a reply, which will be published concurrently when possible.

Sugar, pH, and Strength Changes in Cotton During Storage

U. S. Department of Agriculture
Agricultural Marketing Service
Washington 25, D. C.
January 31, 1958

To The Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

On a selection of samples reported in a recent study [3], measurements [1] were made of sugar content (percentage of soluble reducing sugar) and acid-alkalinity (in pH units). Sugar content of cotton decreases as fibers mature; therefore, a high sugar content may indicate immaturity, as in Tinged cottons, in which cotton bolls may be opened prematurely by frost. (Or it may indicate contamination with honeydew.) As cottons remain in the field and become lower in grade, sugar content usually disappears. Its disappearance may be associated with an increase in pH that results from the action of micro-organisms. As reported by Marsh [2], the pH test offers promise as a rapid method for detecting growth of micro-organisms on cotton.

To study the sugar and pH change under storage in comparison to the color changes, measurements were made on samples stored at 0° and 85° F. at high humidity, and for samples held under two conditions of humidity at 50°, 70°, and 100°.

The distribution of sugar and pH seems to change only at those conditions in which there was considerable microbiological growth. Even at 90% humidity there was no significant sugar or pH change at 100° storage, although the change in color is even greater at this condition than it is at either 85°/high humidity or at 70°/85%.

Because it seemed possible that the very dark samples in this study which resulted from high *Penicillium* contamination might also be weaker in strength, tests for strength were made on a few of the cottons. But no strength loss was found,[2] even for samples that had shown the greatest change in sugar and pH. The strength of samples that were low in color after storage remained about the same whether they had yellowed only in the direction of normal aging or whether they had yellowed and had also darkened because of *Penicillium* mold. If the tests showed any difference in strength it was in the direction of an increase rather than a decrease.

Results for this study for sugar, pH, and strength tests are assembled in the accompanying Figure 1 for comparison. The data show clearly that there are greater changes in the high grades than in the low, and that the only real changes occurred under high humidities at temperatures 50°–85°.

A study of the relation of sugar and pH to color change, particularly for the paired samples at the same temperatures but different humidities, seems to show that only minor differences in sugar and pH

[1] According to methods reported in Cotton Testing Service, Agri. Mktg. Serv. Report No. 16 and in Regulations and Fees for Cotton Testing Service, Agri. Mktg. Serv. **38**: 1–33.

[2] Some types of micro-organisms do cause a change in strength [1].

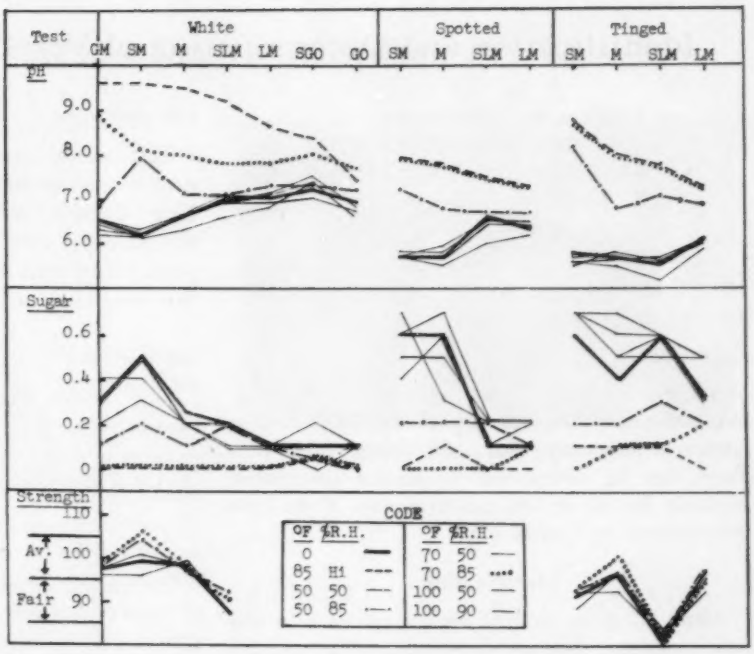

Fig. 1. Sugar and pH tests on cottons after 9 months storage at conditions ranging from 0° to 100° temperature and high (85%–90%) to low (50%) relative humidity, and strength tests (Pressley index) on a few White and Tinged grades that showed wide changes in color, sugar, and pH. The only real changes occurred under high humidities, and there the changes are greatest in the higher grades. No significant changes occur in strength; certainly there is no loss in strength.

occur even for large color changes when the color change is chiefly in the direction of increasing yellowness that is normal to cotton storage, but that there are large changes in both sugar and pH when there is microbiological action in the presence of high humidities. It therefore seems probable that while the change in these factors is closely related to microbiological activity (which sometimes results in a color change, sometimes not), it is not closely connected with the chemical changes involved in the normal yellowing of cotton with age.

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1. Marsh, P. B. et al., TEXTILE RESEARCH JOURNAL **19**, 466–474 (1949).

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3. Nickerson, D. and Tomaszewski, J. J., TEXTILE RESEARCH JOURNAL **28**, 485–497 (1958).

DOROTHY NICKERSON
JOSEPHINE J. TOMASZEWSKI

Identification and Determination of Verel[1] Fiber in Fabrics

Research Laboratories
Tennessee Eastman Company
Division of Eastman Kodak Company
Kingsport, Tennessee
January 31, 1958

To The Editor

TEXTILE RESEARCH JOURNAL

Dear Sir:

A procedure has been developed for the identification and quantitative determination of Verel fiber in fabrics; it is assumed that fibers present other than Verel can be determined by known procedures. Methods for the determination of most fibers have been covered by Luniak [1].

Identification

About 0.2 g. of scoured fiber is placed in a test tube containing 5–6 ml. of pyridine. The mixture is heated on a steam bath for 2–3 min. If Verel fiber is present, it does not dissolve but turns a deep reddish brown; the pyridine becomes pale pink. The amount of color developed depends on the heating time.

Quantitative Determination

The size of the sample can vary considerably. Usually about 0.5 g. of fiber is used. The specimen should be dissected into yarns and these cut into short pieces ($\frac{1}{8}$–$\frac{1}{4}$ in.) and untwisted.

After being dried at 50° C. for 24 hr., the fiber sample is weighed accurately and placed in a 125-ml. glass-stoppered flask containing 50 ml. of the selected solvent. The flask is shaken occasionally over a period of 2 hr. to dissolve the soluble fiber. The fiber remaining undissolved is collected on a tared sintered-glass filter and washed with two 25-ml. portions of solvent. The residual fiber is dried at 50° C. for 24 hr. and weighed.

Acetone

This solvent, employed at 35–40° C., is used to separate Verel (soluble) from any fiber except Dynel, cellulose acetate, and Vinyon HH.

Cyclohexanone

This solvent, employed at room temperature, is used to separate mixtures of Verel (insoluble) and Dynel (soluble) or Vinyon HH (soluble). When this solvent is used, the undissolved fibers should be given a final wash with ethyl alcohol in the separation procedure to speed the drying of the fiber.

Acetic Acid

This solvent, employed at room temperature, is used to separate mixtures of Verel (insoluble) and cellulose acetate (soluble). A final wash with ethyl alcohol is also recommended in this case.

Discussion

The qualitative test described for the determination of Verel fiber is very specific. Only one other known fiber, Saran, develops a red color in pyridine. Saran, however, is soluble in pyridine, whereas Verel is not.

Verel is also characterized by its flame resistance, the sharp acrid odor of its combustion products, and its peanut-shaped cross section.

The procedure described for the quantitative determination of Verel has given good results in these laboratories. Verel fiber was determined in blends within 2% of the true value.

It should also be noted that this quantitative procedure can be used for separating complicated fiber blends by employing the solvents consecutively and filtering after each treatment. For example, a mixture of Verel, Dynel, cellulose acetate, and wool could be separated by first using acetic acid to dissolve the cellulose acetate, following with cyclohexanone to dissolve the Dynel, and then using acetone to dissolve the Verel; the wool is left unaffected.

Literature Cited

1. Luniak, Bruno, "The Identification of Textile Fibers," Sir Isaac Pitman and Sons Ltd., London, England (1953).

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[1] Verel is a registered trademark of Eastman Kodak Co.

A Self-Resonating Vibroscope

American Enka Corporation
Research and Engineering Division
Enka, North Carolina
February 18, 1958

To The Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

The general theory and operating principles of the vibroscope have been discussed in several articles over the past decade. It has proved to be a useful tool in the study of single filament properties. However, there has been a recent development of this apparatus, both in this country and in England, which I feel will be of interest to your readers. In this modification, the fiber denier is indicated on a meter as soon as the fiber is installed in the clamps.

The direct reading vibroscope is achieved by making the fiber a part of an electromechanical oscillator, with the mechanical properties of the fiber determining the frequency of oscillation. In one model the filament is given an electric charge, and acts in effect as one plate of an electric condenser. Any motion of the filament toward or away from the other condenser plate, mounted close by, results in a changing voltage on this second plate. This signal is then amplified and fed to a small speaker to the cone of which is fastened a small pin. Thus the speaker and pin move in step with the fiber, and since the fiber is mounted to rest lightly against the pin, the amplitude of the vibrations tends to increase.

Although random disturbances initially give rise to a variety of frequencies in the fiber motion, the amplitude will be greatest at the resonant frequency. As a result, the frequency at which the entire circuit oscillates is determined by the fiber characteristics. As long as the filament is suspended in position with the proper tension, the circuit will remain in oscillation at the resonant frequency of the fiber. A frequency meter can then be calibrated to read directly in deniers.

Instruments accomplishing this have recently been described in the literature [1, 2]. A photograph of one model developed in this laboratory is shown in Figure 1; Figure 2 is a typical calibration curve. It is quite feasible to extend the range of this instrument beyond that shown, but our requirements have not made this necessary. The model illustrated has been in use in our Research Textile Laboratory for more than a year, and has proved quite satisfactory in both reliability and operating ease.

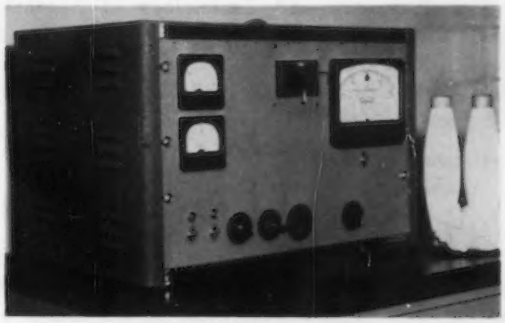

Fig. 1. Self-resonating vibroscope.

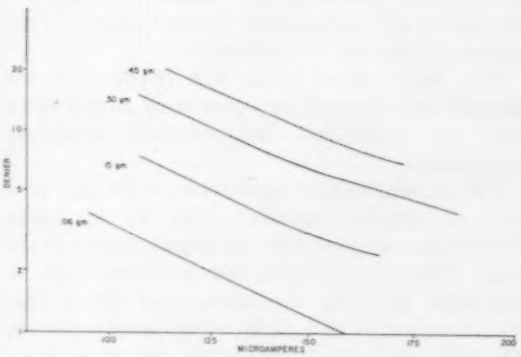

Fig. 2. Denier vs. meter current. Tensioning weight is selected to cover the necessary denier range.

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1. Butler, K. J., *Skinner's Silk and Rayon Record* **32**, 51 (1958).
2. Kemic, C. S. and Parker, J. P., *Rayon Review* **11**, 141 (1957).

J. P. PARKER

Book Reviews

The Chemistry and Technology of Leather, 1956. Vol. 1. F. O'Flaherty, W. T. Roddy, and R. T. Lollar, ed. A.C.S. Monograph No. 134. New York, Reinhold Publishing Corporation, 1956. 495 pages. Price $14.00.

Reviewed by Albert E. Chouinard, Department of Leather Engineering, Lowell Technological Institute, Lowell, Massachusetts

This volume is the first of a series of four volumes designed to cover every technical aspect of tanning and leather manufacture. It has been prepared by 14 distinguished contributors and edited by Dr. Fred O'Flaherty, Mr. William T. Roddy, and Dr. Robert M. Lollar, of the Tanners' Council Laboratory, University of Cincinnati.

This first volume opens with chapters on the physical and chemical constitution of animal skins from the histological and chemical viewpoints respectively.

The macromolecular organization of the skin proteins is discussed separately from the nonprotein constituents and constitutes the largest chapter in the book. This chapter is the best up-to-date body of knowledge regarding the soluble and fibrous proteins of animal skins available to the leather chemist. The bibliography at the end of the chapter is an invaluable source of information to the researcher and the student.

The contributors vary somewhat in their treatment of the subjects; this is quite understandable in view of the unusual combination of theoretical and laboratory results with industrial practice. This is evident by the fact that one chapter is devoted to the technical practice of unhairing and two chapters to the mechanism of unhairing and the chemistry of liming. This emphasis on the chemistry of liming is due to the present-day almost universal use of liming operations to remove the hair and to prepare the skin for tanning.

This distinguishing of practice and theory is again evident in the portion of the book devoted to bating. The procedure, operation, and general effect of bating is handled separately from the history and the chemistry of enzymes used in bating. This tends to give a sense of divorcement and leads to a small amount of repetition or loss of continuity, particularly to the neophyte. However, the definite advantage of having each phase of practice and theory separately developed by qualified individuals offsets all lesser and obvious disadvantages.

Most of the chapters are well documented with references to patents and the technical literature; the bibliographies at the end of each chapter seem to be as complete as is humanly possible and consistent with the nature and available knowledge of the fields covered. The greatest shortcoming naturally lies in the general fields of practice, wherein little has been published or disclosed regarding what was, until relatively recently, a mysterious secretive art.

The successful coordination of the work of the 14 contributors is definitely due to the thoroughness and excellence of the editors. The contributors were well chosen; their work as contained in the 14 chapters is the best compilation of theory and practice on the operation of animal skin treatment, prior to tanning, yet made available to the leather industry. They are to be congratulated for their persistence and courage.

A volume of this type brings home rather forcefully the very broad aspects of leather science and technology in that it indicates the penetration of leather making into so many of the generally distinct basic sciences. This volume has no doubt set a very high standard for the three remaining volumes yet to appear in the near future.

Natural and Synthetic Fibers Handbook, 1956. Milton Harris and H. Mark, ed. New York, Interscience Publishers, 1945. 947 pages. Price $45.00.

This volume comprises the abstracts published as Series II, Volume III of the well known Natural and Synthetic Fibers Literature and Patent Service, Issues 1–12. Far more complete than the usual abstracts, generally containing original data, graphs, photographs, etc., these abstracts frequently make it unnecessary for workers to refer to the original papers. Their availability in loose-leaf form makes them easily accessible in libraries for reference purposes.